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October 2024

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LITHIUM PROCESSING

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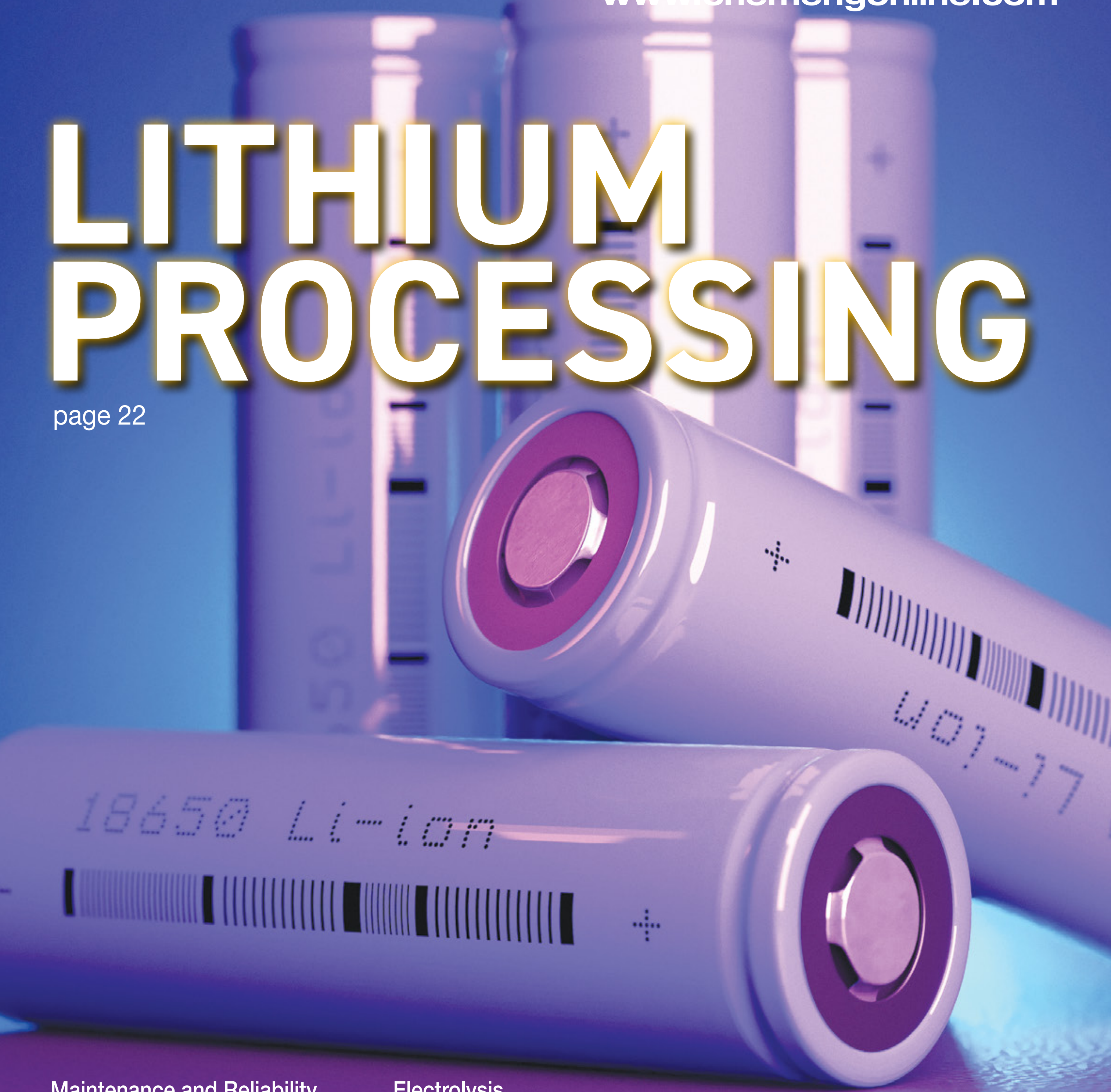

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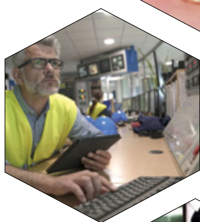
Technical and Practical



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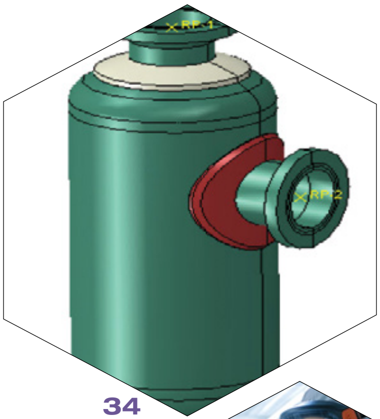

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Editor's Page

Advancing battery technologies

Last month, the U.S. Department of Energy (DOE, www.energy.gov) announced that two Energy Innovation Hub teams would receive a total of $125 million ($62.5 million to each team) in funding over five years to "seed and accelerate next-generation technologies beyond today's generation of lithium (Li)-ion batteries." The two Energy Innovation Hub teams are the Energy Storage Research Alliance (ESRA) and the Aqueous Battery Consortium (ABC).

Energy Innovation Hubs

ESRA will focus on developing compact batteries for heavy-duty transportation and grid energy storage. The ESRA team consists of nearly 50 researchers, led by DOE's Argonne National Laboratory (ANL, www.anl.gov) with both Lawrence Berkeley National Laboratory (Berkeley Lab) and Pacific Northwest National Laboratory (PNNL) as co-leaders. Twelve universities are partnered with the three national laboratories in this effort. ESRA will ground its work in basic science to address challenges in battery development, including achieving safety, high-energy density and long-life using inexpensive, abundant materials. In its press release, ANL says, "ESRA seeks to enable transformative discoveries in materials chemistry, gain a fundamental understanding of electrochemical phenomena at the atomic scale and lay the scientific foundations for breakthroughs in energy storage technologies."

ABC aims to establish the scientific foundation for large-scale development and deployment of aqueous batteries for long-duration grid-storage technologies. Stanford University (www.stanford.edu) and SLAC (Stanford Linear Accelerator Center; www.slac.stanford.edu) will lead the team of 31 scientists, engineers and physicists from 12 universities, SLAC, the U.S. Army and Naval Research Labs. ABC will work toward developing an aqueous battery with a higher energy density than current lead-acid batteries that is environmentally safe and considerably less expensive than Li-ion batteries. Both ESRA and ABC will prioritize using materials that are abundant to avoid supply-chain bottlenecks.

In this issue

Today's rechargeable batteries are dominated by Li-ion technology. The growing demand for these batteries in everything from small electronics to electric vehicles to large-scale energy storage is prompting a surge in the need for lithium. This month's cover story examines the current state of lithium supply and processing in the U.S. (see Lithium Landscape: Activity is Charging Forward in the U.S., pp. 22–26).

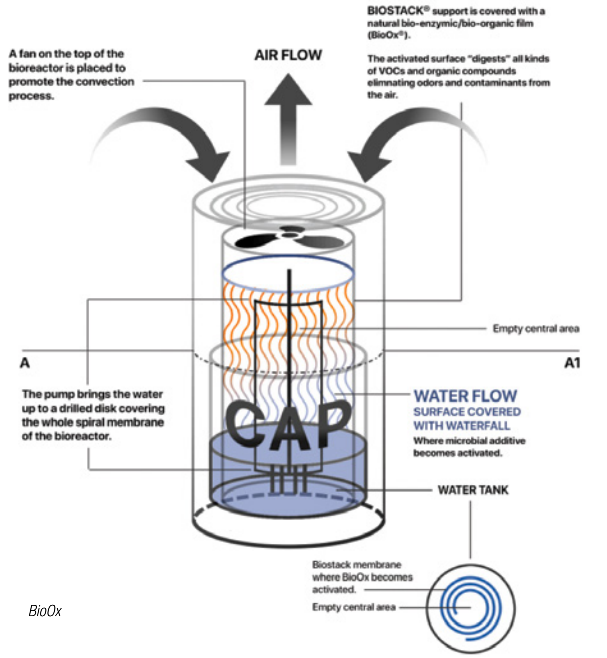
In the meantime, researchers are following multiple pathways to improve battery technologies beyond the Li-ion standard, with goals that include achieving higher energy densities, better safety and cost-effectiveness. One promising pathway is leading toward solid-state batteries (SSB). A number of the challenges involved with SSB development are discussed in our Solids Processing article (Controlling Process Variables for Solid-State Battery Performance, pp. 41–43). Advances in energy storage technologies are surging (see for example Solid-State Battery Advances, p. 9), and *Chemical Engineering* will continue to follow and report those advances. ■

Dorothy Lozowski, Editorial Director

Comprehensive destruction of airborne contaminants in a compact bioreactor

There are many categories of air contaminants that may be encountered in industry, from volatile organic compounds to aerosols to viruses, as well as a range of particulate matter and ultra-fine particles, which also vary greatly in size and properties. This makes it difficult for a single air-cleaning technology to comprehensively handle such contaminant mixtures. The air-cleaning technology developed by BioOx (East Hanover, N.J.; www.bioox.us) claims to capture and destroy a wide array of airborne contaminants using a novel bioreactor system.

"Usually, competing systems absorb, but do not destroy, the contaminant. For example, activated carbon will adsorb hydrocarbons, but then you still must regenerate and recover the hydrocarbons for destruction before re-using the carbon. BioOx can oxidize hydrocarbons to CO_2 and H_2O biologically. This means no regeneration is required," says Sam Sofer, president and founder of BioOx. Within the BioOx reactor, enzymatic media are immobilized on a specialized spiral-biosupport membrane (see diagram). Water is pumped up from the bottom of the reactor, where it flows down the membrane, keeping the media moist. Water is distributed evenly from above the biosupport, exposing contaminants to the enzymes, which are activated to begin bio-oxidation upon water contact. "A fan at the top of the reactor promotes convection, creating a clean air dome. As water flows into the tank, it captures contaminants via Bernoulli's Principle in tiny bubbles. Once the air pollutants are captured, brought into the unit and digested by the media, the clean air dome expands to cover a larger and larger area," says Sofer. The special spiral design of the bio-support membrane supports contaminant contact with the enzymes due to the turbulence created by cyclone action.



BioOx

Sofer says that the BioOx system is especially well-suited for manufacturing areas where quality control is key as product batches change and invisible air contaminants may remain, such as in the production of paint or flavor ingredients. "For example, if you're making red paint, and now you want to make white paint, and there are invisible red paint particles in the air, the quality control will suffer." The BioOx reactor has been demonstrated in a variety of facilities with up to 200 lb/d contaminant destruction.

Recycling mixed medical waste with steam cracking

Currently, medical waste streams are complicated to recycle because these single-use products — such as facemasks, syringes, nitrile gloves and non-woven gowns — consist of multiple materials and must be considered contaminated with potentially infectious particles. Now, researchers at Chalmers University of Technology (Göteburg, Sweden; www.chalmers.se) have made progress toward a viable method for recycling mixed medical waste using thermochemical recycling.

In a study published recently in *Resources, Conservation and Recycling*, the Chalmers team showed that a steam-cracking process could be effective for breaking apart mixed streams of medical waste into its chemical building blocks. Depending on the types of waste and proportions, recycled medical products can yield mixtures of light olefins, ethane, BTX (benzene, toluene, xylenes) and other molecules that could then become feed material for the petrochemical industry to make new plastics.

The team designed a bubbling fluidized-bed reactor that could handle solid material, and heated the material to 700–800°C to initiate a reaction similar to the steam cracking of naphtha. In the reactor, the waste materials are broken into small molecules and microorganisms are destroyed. Through a series of tests, the researchers processed mixtures that approximate hospital waste, containing 10 different plastic types, as well as cellulose.

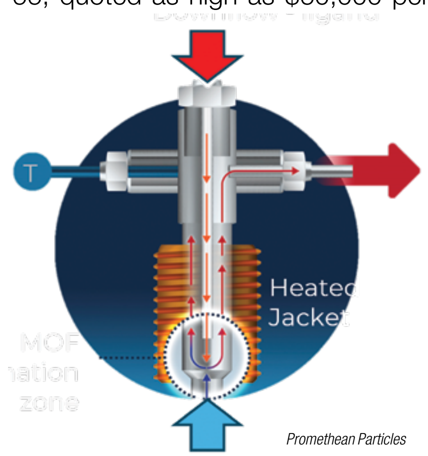
The hospital waste was tested at different temperatures (700, 750 and 800°C) in a laboratory-scale reactor. "A significant portion of the carbon in the feedstock could be effectively recovered as valuable chemical building blocks . . . enabling their direct application in the chemical industry and reducing reliance on fossil resources," the researchers write. "At 700°C, carbon recovery percentages were approximately 79% for face masks, 82% for plastic syringes, 38% for nitrile gloves, and 76% for non-woven gowns, they found.

The Chalmers scientists are currently developing the recycling technology based on an in-house industrial-scale fluidized-bed steam cracker with a capacity of 100 kg/h for various plastic-rich feedstocks together with industrial partners.

Process for continuous production of MOFs plans scaleup

Metal-organic frameworks (MOFs) have shown great potential for a number of applications, including as carbon-capture materials, but their use commercially has been limited by production methods, which are generally expensive batch processes. With the development of a continuous process for producing a range of MOFs at industrial scale, Promethean Particles (Nottingham, U.K.; www.prometheanparticles.co.uk) aims to lower the costs of making MOFs and expand production volumes.

MOFs are a class of materials where metal ions or clusters are linked with rigid organic linker molecules to form highly porous crystalline compounds. "We've seen current prices for the supply of a common MOF material, known as UiO-66, quoted as high as $60,000 per kg by other MOF manufacturers," says James Stephenson, Promethean CEO. "With further development of our continuous-flow process, we think we can reach a target of $30 per kg for our MOF products," he says, adding, "We're trying to change the perception of what is commercially viable when it comes to MOFs."



Promethean Particles

Promethean recently announced funding from an investment round that will be used to build a new facility with the capability to expand their production capacity by 5–10 times, compared to their current capacity of 1,000 tons per year. Production at those costs and volumes would open the door for more widespread use of MOFs in carbon-capture applications, water-harvesting applications in water-stressed regions, and other uses.

In Promethean's process, called continuous flow hydrothermal or solvothermal synthesis (CFHS or CFSS), a solution of metal salts and a solution of organic linkers are combined at a specified temperature in a patented counter-current reactor, which can be operated at elevated pressure. Within the reactor, the two solutions mix in turbulent flow and nucleation of MOF particles occurs (diagram).

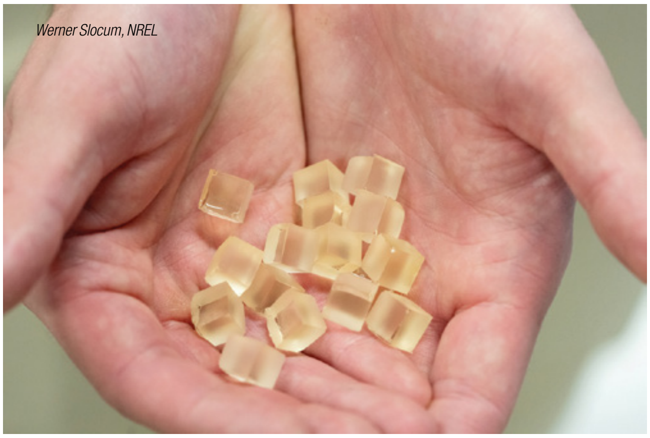
Promethean product manager Selina Ambrose explains that by carefully controlling the temperature and pressure of the reactor, as well as the fluid flowrates, the mixing dynamics can be influenced, meaning the structural properties of the resulting MOF could be controlled. These properties include particle size, shape or crystallinity. The product from the reactor is a suspension of MOF particles in water or other carrier solvent. Once the particles form, the continuous-flow nature of the process allows an option for secondary and tertiary ingredients to be introduced in situ, to further modify the MOFs and obtain the desired properties.

Recyclable wind turbine blades

As wind energy takes a prominent role in the energy transition, researchers at the U.S. Department of Energy's National Renewable Energy Laboratory (NREL: www.nrel.gov) have focused on manufacturing sustainable turbine blades that can be made from bio-derivable resources and then chemically recycled at the end of their useful life. The researchers demonstrated the development with a 9-m-long blade made from a biomass-derivable resin named PECAN (PolyEster Covalently Adaptable Network; photo) that was developed by the NREL using bio-derivable sugars.

The findings are published in the journal *Science*. Nic Rorrer, one of the two corresponding authors of the *Science* paper, said one of the concerns raised in the literature about these types of materials is that the blade would be subject to greater "creep," which is when the blade loses its shape and deforms over time. The testing at NREL showed recyclable materials can be used. Composites made from the PECAN resin held their shape, withstood accelerated weatherization validation, and could be made within a timeframe similar to the existing cure cycle that is currently used in wind-turbine manufacturing. While wind blades can measure the length of a football field, the size of the prototype provided proof of the process.

Under existing technology, wind blades last about 20 years, and afterward they can be mechanically recycled and shredded for use as concrete filler, for example. PECAN marks a leap forward because of the ability to recycle the blades using mild chemical processes. Ryan Clarke, a postdoctoral researcher at NREL and first author of the new paper said the chemical process was able to completely break down the prototype blade in six hours.


Werner Slocum, NREL

The U.S. Department of Energy jointly funded the research through its Advanced Materials and Manufacturing Technologies Office and Bioenergy Technologies Office and their support of the BOTTLE Consortium. Additional research and funding will allow the NREL investigators to build larger blades and to explore more bio-derived formulations.

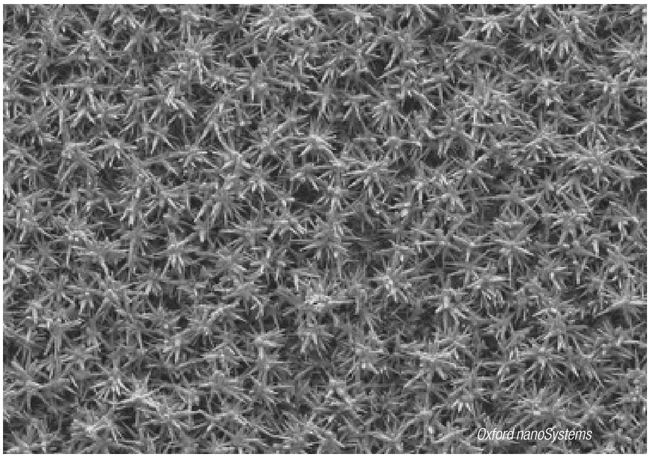
A low-cost coating system designed to optimize water electrolysis

Seemingly small component changes can make a large impact on the productivity of electrochemistry systems, such as electrolyzers used for producing "green" hydrogen. For example, a new coating technology developed by Oxford nanoSystems Ltd. (OnS; Abingdon, U.K.; www.oxfordnanosystems.com), when applied to the electrodes of alkaline electrolyzers, can boost the electrical current flowing through the system, thereby increasing the rate of hydrogen production. "The resulting increase in production capacity lowers the CAPEX cost of alkaline electrolyzers by over 50%, relative to existing technologies. This very substantially reduces the cost of producing green hydrogen," notes Ian Russell, CEO of OnS.

Historically, alkaline electrolyzers have relied on coating materials containing expensive platinum-group metals (PGMs) to enhance performance. However, the OnS coating, nanoFLUX, provides comparable performance to PGM-loaded coatings at a lower cost than conventional nickel-based electrodes, says Russell. Key to this performance is the coating's dendritic structure that creates a network of microcavities on the substrate's surface (photo) that significantly enhances the density of sites available for bubble formation. Additionally, the highly porous nature of nanoFLUX facilitates the release of bubbles and promotes surface re-wetting. This leads to a greater number of smaller bubbles being generated and expelled from the coated surface at an accelerated rate, thereby minimizing resistance and losses due to bubble buildup.

"Bubble release is critical because the electrochemistry reaction takes place where the liquid electrolyte is in contact with the solid electrode. Prior to their release from the electrode surface, gas bubbles form an insulating layer between solid and liquid, reducing the effective area of the electrode," explains Russell.

For electrolyzer applications, the nanoFLUX coating has been combined with a proprietary non-PGM catalyst to activate the hydrogen-generation reaction. "The coating process is based on aqueous electrochemistry. The principal steps are the electroless deposition and growth of the nanoFLUX layer, followed by electroplating of the catalytic later. In addition, there are some surface-cleaning, preparation and rinsing steps within the overall process," says Russell. OnS is currently operating a demonstration plant with the capability to coat electrodes up to 700 mm in diameter — similar in scale to those used in commercial systems — with plans to move to commercial production in 2025.



Oxford nanoSystems

Scaleup underway for super-efficient silver refining

A significant contributor to the cost, energy consumption and carbon footprint of silver production is in the refining steps to remove, recover and recycle zinc from Parkes Crust silver-zinc intermetallic compounds.

A fundamental redesign of the silver process to improve dezincing kinetics — the rate-limiting step in silver refining — was recently reported in the *Journal of Sustainable Metallurgy*. The new Britannia Silver Process (BSP) introduces a redesigned vacuum dezincing unit and removes a liquation stage that requires the crust to be melted and mixed with lead.

"The use of natural-gas heating in the BSP dezincing process compared to electric-induction operation used in current processes delivers significant energy-efficiency improvements. Liquation in the current process is very energy-intensive, and its replacement also delivers significant energy-efficiency gains," explains Steven King, a principal metallurgist with the Glencore Group (Baar, Switzerland; www.glencore.com), who conducted the work along with Alberto Striolo, professor of chemical engineering at University College London (UCL; www.ucl.ac.uk) and University of Oklahoma (Norman; www.ou.edu), at Glencore's Britannia Refined Metals (BRM) facility in the U.K. The work carried out at BRM was conducted with a significant emphasis on process safety with no accidents, despite the experimental nature of the project, notes King.

In pilot operation, the new process has demonstrated dezincing kinetics 44% higher than the original process, as well as a higher purity and recovery efficiency of zinc. "The pilot plant used to develop the process and validate performance metrics was a ¼-scale rendition of a full-scale plant. Development of a demonstration-scale plant is now in progress. I would consider the dezincing geometry we have developed to be new and fully original," says King. The pilot-scale operation also revealed a nearly 40% reduction in energy usage and 31% reduction in Scope 1 carbon emissions, while producing a silver product of comparable quality to traditional refining processes.

Another benefit of the BSP is the promise of recycling and reducing waste, such as slag. "Current processes dispose of this waste stream. The BSP presents the opportunity to recycle the flux and direct the zinc oxide/lead oxide residue to the zinc-recovery processes," adds King.

Scaling up brine electrolysis for high-purity lithium hydroxide

A significant portion of the energy and cost requirements in the production of lithium-ion batteries stems from the cost of converting lithium sulfate or lithium chloride into lithium hydroxide monohydrate (LHM) of sufficient purity for battery use. Noram Electrolysis Systems, Inc. (NESI; Vancouver, B.C., Canada; www.nesi.tech) was awarded funding from Natural Resources Canada (NRCan; www.natural-resources.canada.ca) to support the commercialization of lithium hydroxide production by electrolysis, including the development of a dedicated lithium-electrolysis testing center with extensive brine-treatment infrastructure where the company can use its Norscand electrolyzer to produce battery-grade LHM.

"In 2017, we started running, as far as we're aware, what is the world's first commercial-scale membrane-electrolysis cell to produce lithium hydroxide from lithium sulfate at Nemaska Lithium's P1P demonstration plant in Shawinigan, Quebec, and then at our large-scale pilot plant in Richmond, B.C. Following this, we modified our large-scale pilot plant to operate a commercial-scale membrane electrolysis cell to produce lithium hydroxide from lithium chloride brine as well. Now, with the new testing center to be located nearby, we'll be able to bring in brines and other lithium resources from around the world to this facility and process them into tonnage quantities of LHM, which then can be used for running battery production trials to validate the product and to confirm design criteria for engineering of industrial production facilities," says Jeremy Moulson, president and CEO of NESI.

In a Norscand electrolyzer, lithium solution (anolyte) and the product lithium hydroxide (catholyte) are circulated through the electrolyzer's two compartments. When direct-current power is applied and the current flows through the electrolyzer, the reaction to form lithium hydroxide occurs. "The system can handle both brine and spodumene-based feed inputs, as well as streams from battery-recycling facilities," adds Moulson, also noting that the system can process either a lithium sulfate or lithium chloride feed with minimal changes to the electrolysis cell, and that cells can be customized with more than two compartments as needed for both lithium and other processes.

NESI's electrolyzers are already being put into action processing lithium-chloride brine into high-purity LHM at the Vulcan Energy Resources GmbH (Karlsruhe, Germany; www.v-er.eu) Zero Carbon Lithium geothermal project in Germany, which began commissioning in August. Once full-scale production begins at this site, it will represent Europe's first battery-grade lithium hydroxide product sourced from a European lithium resource. Offtake partners include Stellantis, Umicore, LG Energy Solutions, Renault Group and Volkswagen.

For more on lithium processing, see our cover story Lithium Landscape: Activity is Charging Forward in the U.S., pp. 22–26.

Chementator Briefs

TIRE RECYCLING

The Mitsubishi Chemical Group (MCG; Tokyo, Japan; www.mcgc.com) has demonstrated chemical recycling of end-of-life tires (ELTs) using the coke ovens at its Kagawa Plant (Sakaide City, Kagawa Prefecture). The company is able to feed crushed ELTs as raw material into its coke ovens and produce carbon black again from the tar. According to MCG, this is the first attempt in the world to produce sustainable carbon black from ELTs using coke ovens. The sustainable carbon black produced is said to have the same performance as conventional carbon black and therefore can be used again in new tires. MCG aims to begin marketing sustainable carbon black made from ELTs by March 2026.

SOLID-STATE BATTERY ADVANCES

Researchers led by the Department of Energy's Oak Ridge National Laboratory (ORNL; www.ornl.gov) have advanced the development of solid-state batteries by optimizing a polymer binder to make a strong, thin film for use with sulfide solid-state electrolytes. The researchers successfully made a flexible, solid-state electrolyte sheet that "could at least double energy storage to 500 watt-hours per kilogram," according to ORNL's Guang Yang. Currently available batteries that use liquid electrolytes are associated with safety concerns due to their flammability, thermal instability and potential leakage. The new sheets may allow production of safer, solid-state batteries. They would separate negative and positive electrodes and prevent dangerous electrical shorts while providing high-conduction paths for ion movement.

Plastic polymers currently used in solid-state electrolytes have much lower conductivity than liquid electrolytes. Sulfide solid-state electrolytes offer an ionic conductivity that is comparable to liquid electrolytes currently used in lithium-ion batteries. The goal of the current study was to find a film that was thin enough for ion conduction and thick enough for structural strength. The work was published in a recent issue of *ACS Energy Letters*.

IRON IN CATALYSTS

In a recent analysis of spent catalyst material from fluid catalytic cracking (FCC) units, WR Grace (Columbia, Md.; www.grace.com) found higher levels of iron species as refiners increase the amount of "opportunity" feedstocks they process. Many of these opportunity crudes, such as oil from shale, are laden with iron, which can poison FCC catalysts and reduce performance. Now, Grace is developing a host of catalyst technologies designed to mitigate the harmful effects of iron contaminants and maximize production value for refiners.

Researchers at Grace developed the iron deactivation protocol (Grace-IDP), a method for simulating the deactivation of catalyst material by iron in a laboratory setting. Under FCC reaction conditions, iron, along with other impurities, can form an amorphous silica-alumina phase at the surface of zeolite catalyst particles. The destruction of zeolite at the surface reduces catalyst performance and decreases the unit's yield.

Leveraging information from the IDP, the Grace team addressed iron poisoning in three ways. In one, known as Grace MILLE technology, an extra step in the manufacturing process introduces more macroporosity into the catalyst materials. This optimizes pore size distribution, which helps improve vaporization of the feed molecules and aids diffusion of feed and products. Also, Grace developed novel passivation treatments for the surface of the catalyst material. Finally, the IDP helped catalyst design of existing Grace catalysts to maximize catalyst matrix surface area.

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- OCI GLOBAL
- PPG
- PRAYON
- SK ENERGY

Plant Watch

SK Energy to start commercial production of SAF

October 16, 2024 — SK Energy, a subsidiary of SK Innovation Co. (Seoul, South Korea; www.skinnovation.com), announced the completion of South Korea's first dedicated production line for sustainable aviation fuel (SAF). The new facility leverages an advanced co-processing technology that integrates bio-based feedstocks with traditional oil processing, allowing simultaneous production of petroleum and low-carbon products. A dedicated 5-km pipeline is installed to feed feedstocks continuously into the manufacturing process, enabling continuous SAF production.

BASF inaugurates new plant for alkyl ethanolamines at its Antwerp site

October 12, 2024 — BASF SE (Ludwigshafen, Germany; www.basf.com) has inaugurated a production plant for alkyl ethanolamines at its *Verbund* site in Antwerp, Belgium. This new investment will increase the company's global annual production capacity for alkyl ethanolamines, including dimethyl ethanolamine and methyl diethanolamine, by nearly 30% to over 140,000 metric tons per year (m.t./yr).

Nippon Shokubai to construct new plant for lithium-ion battery electrolyte

October 12, 2024 — Nippon Shokubai Ltd. (Osaka, Japan; www.shokubai.co.jp) is planning to build a new plant for lithium bis(fluorosulfonyl) imide (LiFSI), which is used as an electrolyte for lithium-ion batteries. The new plant, to be located in Fukuoka Prefecture, Japan, will have a production capacity of 3,000 m.t./yr.

Evonik starts up new spray-drying facility for pharmaceutical products in Darmstadt

October 10, 2024 — Evonik Industries AG (Essen, Germany; www.evonik.com) opened a new facility for drying aqueous polymer dispersions of the brand Eudragit at its site in Darmstadt. These polymers are polymethacrylates suitable for use in drug-delivery applications.

Nouryon significantly expands its sodium chlorate capacity in Brazil

October 10, 2024 — Nouryon (Amsterdam, the Netherlands; www.nouryon.com) has commenced operations at its new manufacturing site in Ribas do Rio Pardo, Mato Grosso do Sul, Brazil, which includes the production of hydrogen peroxide, sodium chlorate and chlorine dioxide. The new investment, designed in partnership with a long-term customer in Brazil, significantly increases Nouryon's total sodium chlorate capacity available to the market and supports the growing pulp and paper industry in the region.

Lotte Energy completes new pilot plant for solid-state battery electrolytes

October 6, 2024 — Lotte Energy Materials (Seoul, South Korea; www.lotteenergymaterials.com) completed a pilot plant for the production of sulfide-based solid electrolytes, a key material for next-generation all-solid-state batteries, at its Iksan Plant 2 in Jeonbuk, South Korea. The pilot plant has a production capacity of 70 m.t./yr of solid electrolytes.

MOL Group starts production at green-hydrogen plant in Hungary

October 5, 2024 — MOL Group (Budapest, Hungary; www.molgroup.info) announced that a 10-MW green hydrogen plant, the largest in Central and Eastern Europe, has started production at MOL's Százhalombatta refinery. The facility produces 1,600 m.t./yr of carbon-neutral green hydrogen, which is used for fuel production, reducing the Danube Refinery's carbon dioxide emissions by around 25,000 m.t./yr.

Evonik opens new plant for sustainable emollients at its Steinau, Germany site

October 4, 2024 — Evonik inaugurated its new production plant for cosmetic emollients at its site in Steinau, Germany. These esters are manufactured using an enzymatic process. The new plant will significantly increase Evonik's production capacity, which will cater to the growing customer demand for sustainable cosmetic emollients.

Mergers & Acquisitions

Gevo acquires Red Trail Energy assets in North Dakota

October 16, 2024 — Gevo, Inc. (Englewood, Colo.; www.gevo.com) has entered into a definitive agreement to acquire the ethanol production plant and carbon capture and sequestration (CCS) assets of Red Trail Energy, LLC in North Dakota for $210 million. The acquisition includes existing CCS assets with total sequestration capacity of 1 million m.t./yr, of which 160,000 m.t./yr are currently being utilized, as well as a low-carbon ethanol plant with 65 million gal/yr of capacity.

Methanex to acquire international methanol business from OCI Global

October 9, 2024 — Methanex Corp. (Vancouver, B.C., Canada; www.methanex.com) entered into a definitive agreement to acquire OCI Global's (Amsterdam, the Netherlands; www.oci-global.com) international methanol business for $2.05 billion. The transaction includes OCI's interest in two world-scale methanol facilities in Beaumont, Tex., one of which also produces ammonia. The transaction also includes a low-carbon methanol production and marketing business

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and a currently idled methanol facility in the Netherlands.

Adnoc acquires stake in ExxonMobil low-carbon H_2 project

October 4, 2024 — ExxonMobil Corp. (Houston) and Adnoc (Abu Dhabi National Oil. Corp.; www.adnoc.ae) announced that Adnoc will acquire a 35% equity stake in ExxonMobil's proposed low-carbon hydrogen and ammonia production facility in Baytown, Tex., expected to be the world's largest of its kind upon startup, capable of producing up to 1 billion ft^3/d of low-carbon hydrogen. A final investment decision (FID) is expected in 2025 with anticipated startup in 2029.

Prayon acquires calcium-phosphate company in the U.S.

October 4, 2024 — Prayon S.A. (Engis, Belgium; www.prayon.com) has acquired the U.S.-based company Natural Enrichment Industries (NEI), a producer of calcium phosphate salts. The NEI production sites located in southern Illinois (Herrin and Sesser) complement the existing Prayon production plant of phosphate salts based in Augusta, Ga.

Indorama establishes JV to construct recycling plants in India

October 4, 2024 — Indorama Ventures Ltd. (IVL; Bangkok, Thailand; www.indoramaventures.com) and Dhunseri Ventures Ltd. have formed a joint venture (JV) with Varun Beverages Ltd., PepsiCo's second-largest bottling company outside the U.S., to establish several new polyethylene terephthalate (PET) recycling facilities in India. The joint venture has already begun the construction of two recycling facilities, planned for completion in 2025. The aim is to reach a combined capacity of 100,000 m.t./yr for recycled PET across all the facilities when completed.

PPG agrees to sell silicas business for $310 million

August 29, 2024 — PPG (Pittsburgh, Pa.; www.ppg.com) agreed to sell its silicas products business for approximately $310 million to Qemetica S.A. (Warsaw, Poland), a privately held manufacturer of soda ash, silicates and other specialty chemicals. The transaction includes PPG's precipitated silicas manufacturing sites in Lake Charles, La. and Delfzijl, the Netherlands.

Air Products acquires hydrogen production assets in Uzbekistan

August 26, 2024 — Air Products (Lehigh Valley, Pa.; www.airproducts.com) will acquire Saneg's hydrogen production assets located at the Fergana Oil Refinery in Uzbekistan for $140 million. The acquisition includes a steam methane reforming unit capable of processing either 100% natural gas or liquefied petroleum gas (LPG).

LyondellBasell to acquire recycling technology firm APK AG

August 23, 2024 — LyondellBasell (LYB; Rotterdam, the Netherlands; www.lyondellbasell.com) announced that it entered into an agreement to acquire full ownership of APK AG, a recycling company in in Merseburg, Germany. APK offers a unique solvent-based recycling technology for low-density polyethylene (LDPE). ■

Mary Page Bailey

Eco-Friendly Housekeeping Assists Sustainability Goals

Facility monitoring, LED lighting and low-impact cleaners help reduce carbon footprint at chemical process industries (CPI) facilities

It is not always possible to refine a process to help reduce a facility's carbon footprint; however, embracing some newer trends in more sustainable housekeeping practices presents an opportunity to become more environmentally conscious without impacting production. Here, we look at three developments in eco-friendly facility upkeep — facility monitoring, energy efficient LED lighting and low-impact cleaning solutions — that can provide a nudge in the green direction, while also ensuring safety, which is always the highest priority in the chemical process industries (CPI).

Monitoring and sustainability

Thanks to more advanced sensors and monitoring technologies, there is a growing trend toward using facility and process monitoring to bolster sustainability initiatives directed at improving efficiency and reducing emissions, while also enhancing safety.

Facility monitoring using sensors tied to data acquisition systems allows users to easily access useful data and use the insights to make better decisions regarding sustainability practices, such as reducing emissions, improving process efficiency, lowering CO_2 footprints and lowering utility or other operational costs.

"Many processors are interested in monitoring electrical, water and gas utilities to capture as much savings as possible, while also being environmentally responsible," says Rick Canfield, product engineer at AutomationDirect (Cumming, Ga.; automationdirect.com).

Don Fregelette, vice president of chemical industry marketing at Emerson (Shakopee, Minn.; emerson.com), adds: "There is also a growing trend in the industry to require more monitoring points as corporate initiatives in areas such as energy reduction, decarbonization, safety and controlling more complex unit operations require a better understanding of the process."

According to Fregelette, today's chemical processors are embracing energy solutions that are aimed at improving combustion efficiency and regulatory compliance, including the following:

- Controlling air-to-fuel ratio, monitoring fluegas composition, monitoring oxygen in fluegas and continuous emissions monitoring solutions (CEMS)
- Improving safety monitoring with device diagnostics for more predictive safety monitoring and using remote proof-testing capabilities to extend the time in between required comprehensive proof tests
- Improving operational efficiency, improving plant mass balance and controlling more complex process conditions

Kris Worfe, Endress+Hauser USA (Greenwood, Ind.; us.endress.com) industry marketing manager for the chemical sector, adds: "Monitoring air quality is also critical because it is regulated by both state and federal authorities, and processors must comply with standards set by these agencies for emissions. Additionally, one of the most important utilities to monitor is steam, both generation and consumption, in an effort to make it as safe, reliable and efficient as possible. Measurement technologies can be used to address flow accuracy, energy content, purity and quality concerns" (Figure 1).

If a processor is interested in monitoring utilities, AutomationDirect's Canfield says there are scalable programmable logic controller (PLC) platforms and extensive options for remote monitoring on a desktop or mobile device using internet or cell connectivity and industry-standard secure protocols.

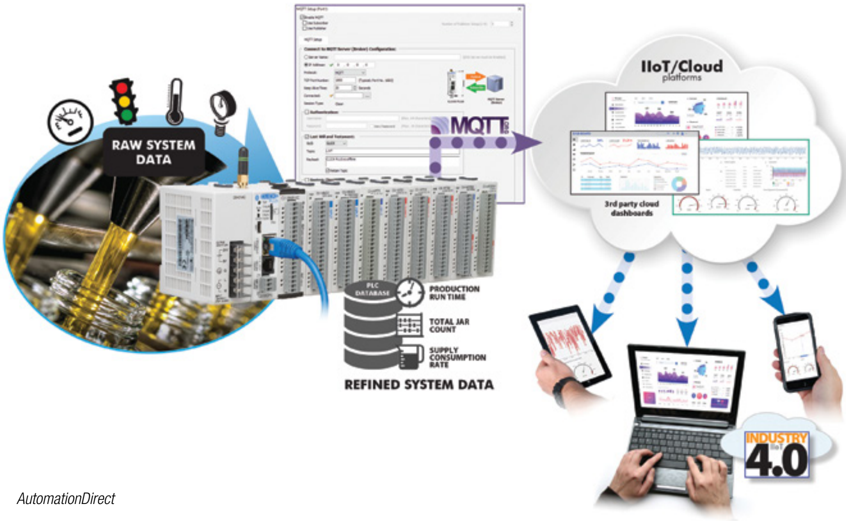
"Once data is transmitted to the cloud, it can simply be viewed or can be integrated into a multitude of analytical systems," he explains. "Users can use this data to optimize their processes and identify inefficient, bro-

FIGURE 1. Wireless connectivity, available for leading modern instrumentation, simplifies maintenance and improves plant safety and efficiency

ken or dangerous processes. A facility monitoring solution doesn't need to interfere with existing core automation processes and can be added in parallel and progressively built out to provide a non-intrusive approach. Users can also incorporate logic or user data entries to the monitoring platform, so it becomes possible to implement direct changes to facility operating parameters for improving efficiency" (Figure 2).

For processors who want to dig a little deeper to improve insight into plant process conditions and control as a means to boost the efficiency and performance of their processes or to double down on sustainability initiatives, Emerson's Fregelette, suggests the use of measurement instrumentation, both wired and wireless. This includes pressure, temperature, level, flow, gas analysis, liquid analysis, flame and gas detection instruments, which can transfer data and asset management information to control systems and automation software (Figure 3).

"Control, safety and data-acquisition systems collect and analyze information from measurement devices in the plant to determine optimal settings," he explains. "That information is used to adjust valves, pumps, motors and drives to ensure product quality, process efficiency, sustainability and safety.



AutomationDirect

FIGURE 2. AutomationDirect offers PLCs, sensors, instruments and networking technologies, making it simple to deploy a non-intrusive facility-monitoring system viewable via the internet or mobile apps

"Further," Fregelette continues, "advanced industrial software, digital twins, process simulation, machine learning and artificial intelligence can empower staff to design, operate and maintain operations for maximum performance and efficiency."

LEDs as a 'green light' solution

"There is currently a trend toward more efficient lighting solutions in the CPI, and LED [light-emitting diode] lights are quickly becoming the technology of choice because LED lighting technology is 40% more energy efficient, more compact and gives off less heat than incandescent lights," says Peter Kohlert, special projects engineer with commercialLEDlights.com (Farmington, Mich.; commercialLEDlights.com).

"LEDs work quite differently from traditional light sources," explains Thomas Geiger, president, LED2work (Chicago, Ill.; us.led2work.com). "They emit light in a 180-degree beam angle, while traditional light sources typically have a 360-degree beam angle. Additionally, heat is generated toward the back of the LED rather than the front, as with traditional light sources. Further, LEDs have a much longer lifespan, potentially lasting up to 60,000 hours.

"In addition, LEDs are significantly more energy efficient, and they produce less harmful waste when recycled. Fluorescent lightbulbs contain mercury, making them problematic for recycling," says Geiger (Figure 4).

But while LED lighting technology offers some major advantages, there are often special considerations for the chemical industry. Geiger mentions challenges such as mechanical stresses from machinery, dust generated from material processing that can settle on lights and lead to increased surface temperatures or degraded performance and the need for protection against water ingress, humidity or gases, as well as chemicals, oils or cleaning agents.

For harsh chemical environments, LED2Work offers LED lighting solutions with an IP 69K rating to provide resistance to dust, water and

Emerson

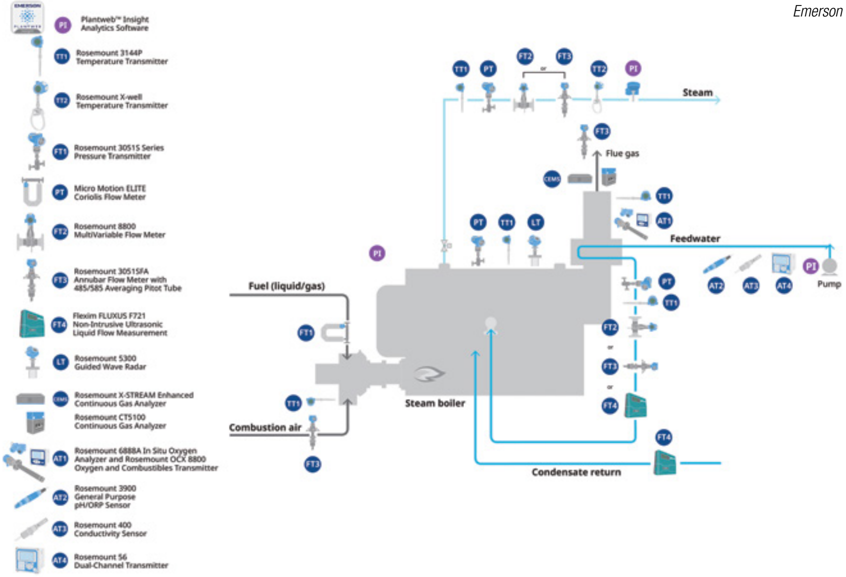


FIGURE 3. Between 30 and 50% of an operating budget is spent on energy. As an example, creating steam is an expensive, energy-intensive process, so evaluating boiler performance offers opportunities to improve energy efficiency and reduce emissions

LED2Work

FIGURE 4. With the latest generation of LED chips, the LEANLED II series from LED2Work provides modern, energy efficient and homogeneous illumination in industrial environments

high-pressure washers, oil and coolant resistance, chemical resistance, impact and vibration resistance and high-temperature tolerance. "These lights are specifically designed for manufacturing environments where standard lighting would fail prematurely," notes Geiger. "In addition to their durability, our lights deliver exceptional light output and are available in a variety of forms and lengths to suit virtually any manufacturing setting. Task-lighting solutions for laboratories and quality control areas are also available to ensure precision and clarity."

Additional challenges found in the CPI include high temperatures of about 50°C, which can result in shortened life expectancy and failed operations, says Neil Peterson, CEO at LED Lighting Supply (Nashua, N.H.; ledlightingsupply.com). For this reason, his company provides an alternative to standard LED lighting "Our long-lasting, UL [Underwriters Laboratories]-rated lighting options are capable of performing in high-temperature areas and have a longer life expectancy than standard LED lighting," says Peterson. "And, our high-temperature, corrosion-resistant lights are designed with quality materials to withstand corrosion and operate in higher temperatures up to 150°C to meet U.S. Occupational Safety and Health Administration (OSHA; Washington, D.C.; www.osha.gov) approval requirements."

Of course, chemical facilities may also have additional requirements due to the presence of hazardous materials, notes commercialLEDlights.com's Kohlert. "Explosion-proof lighting fixtures are designed to prevent ignition sources from causing explosions in hazardous environments," he says.

The major lighting classifications are: Class I Division I (concentrations of vapors, liquids or gases exist within the environment or under typical operating conditions) and Class I Division II (concentrations of vapors, liquids or gases exist within the environment under atypical operating conditions).

Kohlert suggests seeking guidance on hazardous locations from National Fire Protection Association (NFPA) 497 Recommended Practices for the Classification of Flammable Liquids, Gases or Vapors and of Hazardous (Classified) Locations for Electrical Installations in Chemical Process Areas; National Electric Code (NEC) Article 500, which describes the NEC Division classification systems, and Article 505, which describes the zone classification systems; and OSHA 1926.407 Hazardous (Classified) Locations.

That said, the type of activity dictates the type and amount of light needed. "The Illuminating Engineering Society (IES) provides comprehensive guidelines for recommended illumination levels for various industrial locations," says Kohlert. "Regarding explosion-proof lighting, you must understand the nature of the specific hazards as they relate to NFPA 497 for classification."

"Questions to ask include: A) Are there flammable gases, vapors or liquids, combustible dusts or ignitable fibers in the area? B) Are these present under typical operating conditions or under atypical operating conditions? C) Specifically, what types of gases, vapors or liquids, combustible dusts, or ignitable fibers are present in the area?" says Kohlert.

"Before ordering a lighting fixture, questions listed in A, B and C need to be answered. A and C will dictate whether the fixture should be Class 1, 2 or 3. Item B will indicate Division 1 or 2," he explains. "Then there are the questions: What fixture wattage or lumens do you need? How many fixtures and what lighting color? The wattage/lumens are based on the area you wish to illuminate. The color is dependent on the task and somewhat on personal preference or experience. The shape of the room also may dictate the shape of the fixture."

Because lighting is not one-size-fits all and chemical facilities can present complex and unique challenges and may need to adhere to regulatory requirements, Charles Kughn, president of commercialLEDlights.com, recommends a lighting design study, which provides a resource to ensure that facilities get a lighting solution that meets their needs and regulatory requirements, while still maintaining a safe, sustainable and properly illuminated environment (Figure 5).

Greener cleaners

"There is a growing trend toward using more sustainable cleaning products and techniques in the chemical manufacturing industry," says Joshua Schwartz, president and co-founder of Viking Pure Solutions (Port Orange, Fla.; vikingpure.com). "This shift is driven by various factors, including regulatory pressures and the increasing awareness of the environmental and health impacts of traditional cleaning methods."

He continues to say that governments and regulators are now enforcing stricter environmental laws and standards, so chemical processors are required to reduce emissions, minimize waste and improve overall environmental performance, leading to the adoption of more sus-

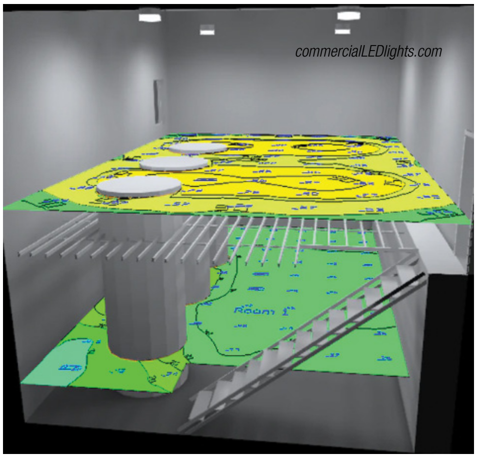


FIGURE 5. A lighting design study, offered by commercialLEDlights.com, provides a resource to ensure that chemical facilities get a lighting solution that meets their needs and regulatory requirements, while still maintaining a safe, sustainable and properly illuminated environment. Shown here is a design study for a chemical mixer

FIGURE 6. Viking Pure solutions are created through an electrochemical process that uses salt, water and electricity to produce a cleaning solution (sodium hydroxide) and a disinfecting solution (hypochlorous acid). Both are effective, safe and environmentally friendly

tainable cleaning practices. In addition, as consumers become more aware of what sustainability means in a broader sense, companies are setting more ambitious sustainability goals, often linked to reducing their carbon footprint, water use and waste generation. "Sustainable cleaning practices help achieve these goals," he says.

Aaron Martin, regional account manager with PathoSans Technologies from Spraying Systems (Glendale Heights, Ill.; pathosans.com), adds: "A more recent development driving new, more sustainable cleaning products is they are proving more efficient — not only providing improved working safety and protecting the environment, but providing an operational advantage. A true win-win!"

Among the choices for effective, yet sustainable, cleaning solutions are electrolyzed water systems that generate cleaning and disinfecting solutions, which are offered by both Viking Pure and PathoSans.

"Viking Pure solutions are created through an electrochemical process that uses salt, water and electricity to produce two distinct products: a cleaning solution (sodium hydroxide) and a disinfecting solution (hypochlorous acid)," says Schwartz. "Both are effective, safe for use around people and equipment and environmentally friendly" (Figure 6).

Produced through on-site generation systems, the cleaning solution is a highly effective cleaner that can break down grease, grime and organic residues. It's suitable for cleaning floors, walls, equipment and other surfaces where chemical residues may accumulate and, because it is non-toxic and non-corrosive, it is safe for workers and reduces the risk of damaging sensitive equipment.

Similarly, PathoSans offers on-site electrochemically activated (ECA) generators that allow users to generate eco-friendly cleaners and disinfectants on site and on demand without the storage of hazardous chemicals (Figure 7). "These chemicals can and have been used within chemical processing facilities for a multitude of different applications, from CIP processes, cross-contamination prevention, equipment and machinery wash down and odor control to the offices where the operators work," says Martin. "Implementing PathoSans chemicals into routine cleaning and disinfection systems has helped reduce the overall carbon emissions, lowered the demand on wastewater management and improved overall safety."

In addition to being effective while also reducing greenhouse gas emissions when compared to the use of traditional cleaning processes, the use of on-site generation reduces the need to manage chemical shipments and storage and reduces the demand on personal protective equipment, as well as the need for hazardous chemical disposal, which provides an operational advantage to any cleaning process, says Martin.

Alternatively, Cold Jet (Loveland, Ohio; coldjet.com), an original equipment manufacturer (OEM) for industrial equipment used to produce dry ice and dry ice blasters, uses recycled CO_2 that has been captured from a carbon capture and utilization process to manufacturer and blast dry ice. "Dry ice is an environmentally sustainable, water-free and chemical-free solution that replaces other harmful processes," explains Steve Wilson, director of dry ice cleaning applications and ESG, with Cold Jet. "It is dry, non-abrasive and does not cause corrosion. Because it is dry, it can be used around electronics and other sensitive components where cleaning with water or other solvents would be problematic.

"Dry ice [solid CO_2] is putting recycled CO_2 to work and is an environmentally responsible solution that does not add additional CO_2 to our environment," he continues. As a matter of fact, both the Environmental Protection Agency (EPA) and California Air Resource Board (CARB) have noted: "Due to the fact that dry ice is recycled CO_2, it will not contribute to your greenhouse gas score. In the calculation of a carbon footprint, the CO_2 is accounted for at the producer level. It is not counted a second time at the point of use." Currently the EPA states that dry ice blasting does not need to be reported under the Greenhouse Gas Reporting Program (GHGRP, 40 CFR Part 98).

"Dry ice uses a gas that is found naturally in our environment," explains Wilson. "Cleaning with dry ice replaces traditional cleaning methods, which often have a higher carbon footprint and often contain human-made fluorinated gases."

Whether driven by regulatory requirements to reduce emissions, the desire to improve overall operating efficiency to minimize costs and waste or corporate initiatives to enhance their brand image, chemical processors should consider incorporating any, or all, of these housekeeping-related strategies to assist with their sustainability goals. ■

Joy LePree

FIGURE 7. PathoSans offers on-site electrochemically activated (ECA) generators that allow users to generate eco-friendly cleaners and disinfectants on site and on demand without the storage of hazardous chemicals

Compressors, Fans and Blowers

Kaeser Compressors

This compressor extends horsepower and flow range

The newly launched SFC 110M variable-frequency-drive compressor (photo) extends the SFC product series to 150 hp, with a 110-kW permanent-magnet motor, and enhances the flow range up to 742 ft^3/min at 100 psig. The SFC 110M features a new Sigma Profile airend, driven by a permanent-magnet motor. This combination boosts energy performance and flow with a smaller physical footprint and less heat, the company says. The new model inherits all the features of the products currently in the series, such as the speed-controlled fan with a brushless direct-current (d.c.) motor for better cooling, and the Sigma Control 2 for superior condition monitoring and external communication capabilities. Other key features of the SFC110M include: improved energy performance, with flow increases up to 20% and specific power below 18 kW/100 ft^3/min; improved maintenance access for faster service, including fluid changes and valve maintenance; optional integrated refrigerated dryers; and reduced environmental impact from lower energy usage. — *Kaeser Compressors, Coburg, Germany*
www.kaeser.com

Alleima AB

New stainless steel for compressor valves

Global demand for cooling systems is rising due to population growth, urbanization, higher living standards, and climate change, and using more energy-efficient cooling systems is crucial. The Freeflex Versa (photo) is a new martensitic stainless steel engineered with a focus on catering to the unique requirements of reciprocating and linear compressor technologies. Notable features of Freeflex Versa include exceptional fatigue resistance, outstanding wear resistance, and the ability to contribute to noise reduction and downsizing. Building upon the success of its predecessors Hiflex and Freeflex Core, the Freeflex Versa compressor valve steel provides increased design freedom, leading to more energy efficient appliances and sustainable products, the company says. The new compressor valve steel is ideally suited for a variety of applications, including air conditioning equipment, heat pumps, refrigerators, freezers and clothes dryers. — *Alleima AB, Sandviken, Sweden*
www.alleima.com

Atlas Copco Airpower n.v.

Battery-powered screw air compressor

Unveiled in May 2024, this company's battery-powered screw compressor, known as B-air (photo), is ideal for remote sites where access to grid electricity is not available. With power delivered from its onboard battery storage, a fully charged B-air can work all day without the need for fuel or a local power source. There are no emissions from the unit and the company says it is surprisingly quiet, with 61 dB measured at 7 m. — *Atlas Copco Airpower n.v., Aartselaar, Belgium*
www.atlascopcogroup.com

This oil-free screw compressor aims for low cost of ownership

The SO-3 oil-free screw compressor (photo, p. 20) achieves peak delivery rate values at lower specific power consumption and has lower total costs over its full lifecycle, according to a total-cost-of-ownership evaluation. The SO-3 has further advantages, the company says, including a large control range, quiet operation, low maintenance and the option to recover heat from hot water temperatures of up to 90°C. The energy efficiency of the SO-3 is said to be best in class, with a wear-resistant design and an integrated IE4 drive motor. The efficiency is further increased by the cooling system, which has low compression and output temperatures. And although the screw compressor is compact, the components are easily accessible for maintenance purposes, the company says. Specific sound insulation

BOGE Kompressoren Otto Boge GmbH and Co. KG

Jenny Products, Inc.

GEA Group

Gardner Denver Inc.

measures have been implemented to effectively reduce noise levels. The compressor is available with frequency control and heat recovery, to increase efficiency. — *BOGE Kompressoren Otto Boge GmbH and Co. KG, Bielefeld, Germany*
www.boge.de

Vertical-tank compressors that are suited to high pressure

This company offers a line of electric two-stage, vertical-tank stationary air compressors that are especially suited for applications requiring greater pressure. The two-stage models (photo) provide 175 psi and displace between 9.1 and 43.6 ft^3/min. Each of the line's 16 models encompasses a smaller footprint than its horizontal-tank counterpart. Each compressor uses a heavy-duty, two-stage cast-iron pump and powder-coated, ASME-certified tank. A large flywheel provides for extra-cooling and easier startup, while special unloading valves further assist the motor in starting. A directional air shroud helps reduce pump temperatures, and a thermal-overload protection feature prevents the motor from drawing too much current and overheating. Tank sizes include 60, 80 and 120 gallons. An automatic start/stop control with a pressure unloader helps to maintain consistent pressure levels, although a constant-run feature can be exchanged for those needing a continuous, heavy flow of compressed air. — *Jenny Products, Inc., Somerset, Pa*.
www.jennyproductsinc.com

This screw compressor is designed to maximize efficiency

With a design for maximizing energy efficiency, the Grasso M is a screw compressor for industrial and marine applications delivers significant savings in total cost of ownership. The M models (photo) consume an average of 3–5% less drive energy than previous screw compressor units. Further, the Grasso M compressors are operated without an oil pump. For refrigeration system operators, this means lower energy and spare parts costs, the company says. The energy efficiency of these compressors is based on the stepless capacity control in conjunction with the extended, variable internal-volume ratio range. The variable speed from 1,000 to 6,000 rpm enables a wide range of applications and maximum efficiency at full and partial load. The sophisticated design and low parts complexity combine reliability and ease of servicing with high efficiency. This saves considerable energy, service and other costs, also contributing to a reduction in the overall operating costs. — *GEA Group, Dusseldorf, Germany*
www.gea.com

Compressors designed for demanding applications

This company's process gas compressors are designed for the most demanding and complex applications up to 1,000 bars (14,500 psi) (lubricated) and 450 bars (6,530 psi) (non-lubricated) discharge pressure. The customized reciprocating compressors are optimized to the user's process conditions in order to achieve longest possible lifetime and highest level of reliability, the company says. Leveraging in-house valve technology, the compressors are designed for maximum durability and long lifetimes. The systems are suitable for use in the gas gathering and processing, gas transport and storage, petroleum refining, chemical and petrochemical sectors, as well as in the industrial gas and hydrogen mobility and energy sectors. — *Burckhardt Compression AG, Winterthur, Switzerland*
www.burckhardtcompression.com

This rotary-vane compressor has a TEFC motor

Hydrovane V/VR series Tank-Mounted Simplex Compressors (photo) are air compressors offering 5–10 hp, 100–150 psi and up to 36.4 ft^3/min. Tank mounted with a totally enclosed, fan-cooled (TEFC) motor, these rotary-vane compressors are engineered to meet stringent specifications. Their compact, simple, robust and integrated design allows them to be fitted anywhere undercover. Ideal for 100% duty cycle applications, they are easy to install and maintain, as well as extremely quiet (as low as 65 dBA). These models also offer configuration flexibility. The company says the performance of the Hydrovane models improve over time once parts are bedded within the rotary stator unit. — *Gardner Denver Inc., Quincy, Ill.*
www.gardnerdenver.com ■

Scott Jenkins

Industrial Electrolysis

Department Editor: Scott Jenkins

Industrial electrolysis involves passing a direct electric current through an electrolyte to elicit a range of chemical reactions. Electrolyzers are key pieces of technology for producing many important industrial chemicals. This one-page reference reviews the basic general operation of an electrolyzer and briefly describes several industrial processes for which electrolysis is a critical part.

Electrolyzer operation

Electrolysis refers to the process of using an electric current to decompose a compound into its constituents. All electrolyzers have an electrolyte, which contains the compound that is to be decomposed and separated, as well as a cathode, from which current flows into the electrolyte, and an anode, into which current flows from the electrolyte. When a direct electric current is applied across the electrodes from an external power source, the ions in the electrolyte are attracted to the oppositely charged electrode (cations are attracted to the negatively charged cathode; anions to the positively charged anode) where they undergo oxidation-reduction (redox) reactions. In redox reactions, the cations gain electrons and are reduced, while the anions lose electrons and are oxidized.

Industrial electrolysis applications

Many important industrial chemical reactions use electrolysis. Listed here are several key electrolysis processes.

Chlor-alkali. The chlor-alkali industry depends on the electrolysis of aqueous sodium chloride (NaCl) to produce sodium hydroxide (NaOH), diatomic chlorine gas (Cl_2) and hydrogen (H_2). Currently, 95% of the estimated 580 chlor-alkali plants around the world use either membrane technology (83%) or diaphragm technology (12.5%) to keep the products separated [*1*]. Brine flows into a series of electrolytic cells and direct-current (d.c.) electricity flows between the electrodes, which are submerged in the brine. Chlorine gas bubbles up through the brine, where it is collected, while NaOH is collected from the bottom of the cell. H_2 is also collected.

Electrolytic purification of copper. For efficient transmission of electricity through copper wires, the copper must be 99.99% pure. An electrolysis process known as electrowinning can be used to carry out this purification. An electrical current passes through an inert anode (positive electrode) and through the copper solution from a prior step in the process, which acts as an electrolyte. Positively charged copper ions come out of solution and are plated onto a cathode as pure copper.

Electrolytic extraction of aluminum from bauxite. Bauxite is a mixture of hydrated aluminum oxides, along with impurities such as iron oxides, titanium oxides and silicon oxide. The last stage of the purification process involves electrolysis of purified aluminum oxide. The aluminum oxide is contained in a solution of molten cryolite, a natural fluoride mineral that is manufactured artificially. Electricity is applied across the electrodes in rows of reduction cells. Inside the cells, high temperatures and a conductive environment, created by the cryolite, breaks down the bonds between aluminum and oxygen [*2*]. The aluminum settles at the bottom of the cells, while the oxygen combines with carbon to make carbon dioxide.

Hydrogen production

An increasingly important electrolysis application is the production of hydrogen gas and oxygen from water. There are three main types of water electrolyzers for hydrogen production:

Polymer electrolyte membrane (PEM) electrolyzers. In PEM electrolyzers, water molecules are electrochemically split at the anode into oxygen gas, hydrogen ions (protons) and electrons (Figure 1). The electrons flow through an external circuit while the protons selectively move across the PEM to the cathode. At the cathode, hydrogen ions combine with electrons from the external circuit to form H_2. The electrolyte membrane is a solid, specialty-plastic material.

U.S. Dept. of Energy

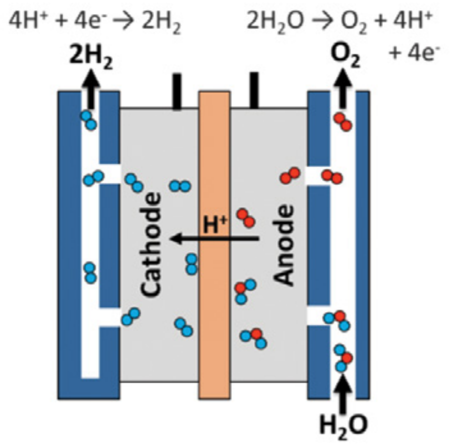


FIGURE 1. In a PEM electrolysis cell, such as the one shown here, hydrogen gas is generated at the cathode

Alkaline electrolyzers. Alkaline electrolyzers operate via transport of hydroxide ions (OH^-) through the electrolyte from the cathode to the anode with hydrogen being generated on the cathode side. Electrolyzers using a liquid alkaline solution of sodium or potassium hydroxide as the electrolyte have been commercially available for many years. Newer approaches using solid alkaline exchange membranes as the electrolyte are showing promise at the laboratory scale [*3*].

Solid oxide electrolyzers (SOEs). Solid oxide electrolyzers operate at elevated temperatures. SOEs combine steam and electrons from the external circuit to form hydrogen and negatively charged oxygen ions (O_2^-) at the cathode [*4*]. As the electrolyte, a solid ceramic material selectively conducts O_2^- to the anode, where it reacts to form oxygen gas and generate electrons for the external circuit. ■

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Lithium Landscape: Activity is Charging Forward in the U.S.

Across several regions of the U.S., new lithium projects are emerging to build up a more localized supply chain

Despite growing demand in the U.S. for batteries in consumer electronics and electric vehicles (EV), the vast majority of lithium extraction and processing occurs overseas, with most extraction taking place in Australia (from spodumene ore) and South America (from brines), and nearly all processing and production activities occurring in China. Recent federal incentives have encouraged the development of a U.S. lithium supply chain, which has spurred a number of new projects and much research and development, all in the interest of promoting U.S. lithium resources and expanding domestic processing and recycling capacities.

Outlook and incentives

"Considering where we were four years ago in the U.S., I feel really optimistic about where we'll be in two years. I am also optimistic that our percentage of global demand is going to increase, which to me is a key metric. We do have an excellent innovation landscape here, from national labs to private industry," says Samm Gillard, co-founder and executive director of the Battery Advocacy for Technology Transformation (BATT) Coalition (www.battcoalition.org). BATT's members include battery experts spanning the entire value chain, from materials extraction through component manufacturing and recycling, as well as advanced materials science, all of whom are interested in incentivizing the domestic production of lithium-ion battery (LIB) materials.

Despite a significant increase in announced lithium projects across the U.S., there are still challenges to overcome in order to move toward commercial-scale production, the most significant being the current low cost of lithium. "If lithium prices are where they are today, it's going to be a challenge to scale up and to get capital in this climate, and also have viable businesses in the U.S. The big question on the processing side is who's going to capitalize at the right time," says Gillard. He further emphasizes that at this point in the economy, companies who can utilize their full capacity effectively will be at an advantage even over those with access to more sophisticated technology assets. "There are a lot of companies that are on the cusp of that. Hopefully the next round of capital from the Bipartisan Infrastructure Law will unlock some of that potential," he adds.

FIGURE 1. A rendering of Stardust Power's planned lithium-refining facility shows the scale of the site, which could significantly increase U.S. domestic capacity for lithium processing

These incentives, coupled with the energy transition encouraging companies to diversify, has led to energy majors like ExxonMobil and Equinor — both of whom have announced investment in lithium production at Arkansas' Smackover formation — to join more traditional electronics conglomerates like Panasonic and Samsung as players in the U.S. battery supply chain. "It's interesting to see these monoliths start to get into the lithium space because the economics are favorable. I think it's a good thing to have companies involved who aren't completely dependent on the dynamics of one particular market," says Gillard.

Midstream refining capacity

Once lithium has been extracted, whether from brine, hard-rock mining or battery recycling, it must undergo a series of refinement steps to prepare it for battery processing. At this point in the lithium value chain, location is key. Currently, China significantly dominates the lithium refining market, but new electric-vehicle tax credits avail-

FIGURE 2. Green Li-ion's plant in Oklahoma started up this year, and plans are in place to quadruple production capacity in the coming years

able under the U.S. Inflation Reduction Act provide benefits for a more domesticated supply chain.

Stardust Power Inc. (Oklahoma City, Okla.) has announced plans to construct a strategically located lithium refinery in Muskogee, Okla. (near Tulsa). "There's a lot of lithium in the U.S. — potentially up to 14% of the global market — but very little battery-grade lithium produced domestically. A critical gap in the supply chain is midstream refining capacity," explains Roshan Pujari, Founder and CEO Stardust Power.

The Stardust Power project is currently one of the largest lithium refineries in the U.S. under development, with a planned capacity of up to 50,000 metric tons year (m.t./yr), to be brought onstream as two 25,000-m.t./yr processing lines (Figure 1). This capacity, says Pujari, could account for up to 10% of future lithium-processing demand in the U.S.

He likens Stardust Power's approach to the lithium market to the oil-and-gas industry where there are producers and refiners in a "hub-and-spoke" model, without necessarily requiring vertical integration across a single organization or site. "The innovative approach that we take is optimizing the large central refinery for multiple sources of lithium, so we can aggregate supply and quickly scale manufacturing of battery-grade lithium. That way, we're not dependent on a single asset," he adds. Another benefit of the U.S. market being primarily in the brine space, is the flexibility that direct lithium extraction (DLE) processes afford in the upstream removal of impurities. "The advantage of DLE using brine is that if there is an impurity that our central refinery cannot accommodate, that can be removed upstream in the DLE process," notes Pujari.

"Being located centrally in the U.S., our shovel-ready site is already connected to major highway, railway and the largest inland waterway system in the U.S. and takes advantage of low-cost renewable power in the form of solar and wind power. We're designing fully electric processing trains to limit air emissions, and we're using zero-liquid-discharge technology, so there's no water pollution. In fact, our byproduct is largely salt," says Pujari.

Stardust Power currently has exclusive options to acquire interests in several lithium-brine projects in the U.S., including Jackpot Lake in Nevada and Pilot Valley in Utah. The company recently awarded a contract to engineering firm Primero USA (www.primero.com.au/northamerica/) to complete a front-end loaded level-3 engineering design and cost study for the facility, after which detailed engineering and procurement will take place.

DLE will be dominant

As the lithium supply chain in the U.S. evolves, it is clear that DLE technologies are a key element in making the most of the available lithium, much of which is incorporated into brine, rather than hard rock.

"Unequivocally, DLE is the way of the future for brines in the U.S. Evaporation ponds have really low recovery rates, and it takes a long time for lithium to go through the process. And in many places, they simply aren't viable without the right amount of sun or with too much precipitation," says Teague Egan, CEO of Energy Exploration Technologies Inc. (EnergyX; Austin, Tex.; www.energyx.com). Taking climate and terrain into account is important as new projects emerge across the U.S. A major area of activity is in the Smackover formation in Arkansas, and neighboring areas of Louisiana and eastern Texas, where the land is much hillier and more forested than the desert regions in South America where brine-evaporation ponds are more common. "This region has some of the best discovered lithium deposits in the U.S. EnergyX's view is that the Smackover shows the highest probability of success and has the most favorable brine characteristics in the country," adds Egan.

With the variation in brine makeup across regions, there are many factors to consider in determining a brine's promise for lithium extraction, such as lithium concentration, impurity profile, temperature and whether processing the brine requires freshwater or other chemical inputs. "Our first goal in designing DLE technology was that we wanted to design a platform that could essentially treat any brine in the world. Our second goal was to develop an end-to-end system, like a petroleum refinery. There's not just one technology in an oil refinery, everything goes through several separation and purification steps to get to usable products. And the same is true of lithium — going from a brine to battery-grade lithium product isn't just one technology. That's why we've pursued a holistic view that incorporates several technology pillars to complete the process," says Egan. For more on EnergyX's DLE technology, read "Lithium Extraction: Prime Time for Brine," *Chem. Eng.*, June 2023, pp. 13–16.

EnergyX recently announced a major lithium project in the Smackover area, Project Lonestar Lithium, which has an anticipated initial processing capacity of 5,000 tons/yr, scaling up to 25,000 tons/yr in a subsequent phase. Kiewit Corp. (Omaha, Neb.; www.kiewit.com) is conducting front-end engineering plans for the project currently.

"We're very confident in our DLE technology, so much so that we've taken the step to start acquiring resources so that we can vertically integrate that technology into our own projects, as opposed to just licensing it to existing lithium producers," says Egan.

Large-scale demonstration

In September, SLB (Houston; www.slb.com) announced a breakthrough in its first sustainable lithium-production project, located in Clayton Valley, Nev. At the site, a 100-gal/min demonstration plant integrates lithium extraction from brine with impurity treatment and concentration technologies to yield high-purity lithium carbonate or hydroxide. "We definitely understand that if we want to meet lithium demand, we can't just rely on the current reserves that in a few hard rock mines in Australia and brine that's coming mainly from Chile and Argentina. So the questions are how can we open up these reserves that have lower concentration, and how can we deal with impurities? This is why DLE technologies keeps popping up, since they are able to handle different kinds of brines. DLE, however, is just the engine, you still need to consider pre-treatment and post-processing requirements," says Nicholas Lugansky, head of mining at SLB.

This is why, in the development of its lithium-production technology, SLB wanted to investigate the entire chain from well to product, bringing in its expertise in subsurface reserve modeling with the development of DLE and other process technologies. "We set out to create an entire flowsheet, starting with the engineering work on how to handle the brine when it comes out of the ground and how to pre-treat it so it can go into the DLE system. We then deploy secondary impurity removal to treat any residual impurities before concentrating the lithium chloride so it can either be shipped as an intermediary or converted into lithium carbonate or hydroxide," explains Lugansky.

The significance of the Clayton Valley site is not only its size — which Lugansky believes is the largest end-to-end lithium-processing demonstration unit currently in operation — but also its focus on sustainable resource utilization. The site was designed to not require any external water source besides the brine, and also to minimize the consumption of reagents and other chemicals, using them for pH adjustment and nothing else. "Taking balance of plant into consideration, we wanted to make sure to address the water question. If you look at where the lithium resources are today, most are in very arid environments, such as Nevada, Utah and California, or desert environments of Chile and Argentina," says Lugansky.

SLB says that its holistic lithium-production technology is 500 times faster than conventional evaporation methods while producing a verified recovery rate of 96% lithium from brine. SLB is currently developing front-end engineering for the entire process flow at the commercial scale, designing for customer parameters so that the units can be fabricated and delivered to customer sites. Lugansky emphasizes that the systems are designed to be modular, standardized and easily replicable for rapid installation at customer sites. "Customers are currently testing our systems at their sites. We hope to announce the first commercial deployment in the next year or so," he notes.

Battery recycling

To augment the demand for "virgin" lithium and other battery metals, and to help make the battery supply chain more circular, new recycling capacity is coming onstream and novel technologies are emerging. In April, Green Li-Ion Ltd. (Singapore; www.greenli-ion.com) opened its first site, a commercial-scale lithium-ion battery recycling plant in Atoka, Oklahoma (Figure 2). The plant is currently designed to process around 700 tons of black mass, equating to a processing capacity of about 2 ton/d, and scaleup plans are already in process. "What we're looking to do in 2025 is support the growing battery materials industry in North America by producing high performance green battery material at scale," says Lucas Marks, vice president of Business Development — North America at Green Li-Ion.

Green Li-ion's Hydro-Rejuvenation™ recycling process is designed for modularity, meaning that battery companies can either provide their own materials to be processed at the site, or manufacturers can license the technology to be installed on their own premises for onsite processing of spent battery materials. The process includes a proprietary reactor design where precursor cathode-active material (pCAM) and mixed hydroxide precipitate (MHP) are produced along with lithium carbonate and graphite, eliminating two metal-refining steps from typical hydrometallurgical recycling processes. The plant includes an onsite quality-control laboratory to test customer materials and also ensure that its produced materials are "drop-in ready" for manufacturing new batteries. The plant is designed to minimize waste and effluent streams, and the only byproducts are solid sodium sulfate and water, which are reusable and saleable product streams. "We've worked with third parties to show that our sustainability numbers are not only a bit better than using virgin materials for pCAM, but also when compared to other hydrometallurgical recycling processes," says Marks.

The company is taking advantage of its strategic location in the central U.S., working with domestic suppliers for spent battery materials to help move the supply chain away from a dependence solely on virgin materials and build the infrastructure required to make batteries more circular. "We're moving in that direction. There are companies who are involved in picking up used batteries to help establish the supply chain. There are quite a few players that are doing upstream black mass production, but to keep and use these critical battery materials in the U.S., we must refine and produce cathode and anode precursors locally, and that's where we come in," adds Marks.

Kevin Hobbie, Global operations manager at Green Li-ion emphasizes the significance of the plant's location within the lithium supply chain: "Oklahoma is uniquely positioned for this. The state has black-mass producers and a company that's converting MHP back to sulfates, as well as the Stardust lithium-production facility that's going to be built about 30 minutes north of our site. And of course, there is the Panasonic battery gigafactory being built in Kansas."

Battery-recycling firm Blue Whale Materials LLC (Washington, D.C.; www.bluewhalematerials.com) is also constructing a processing facil-

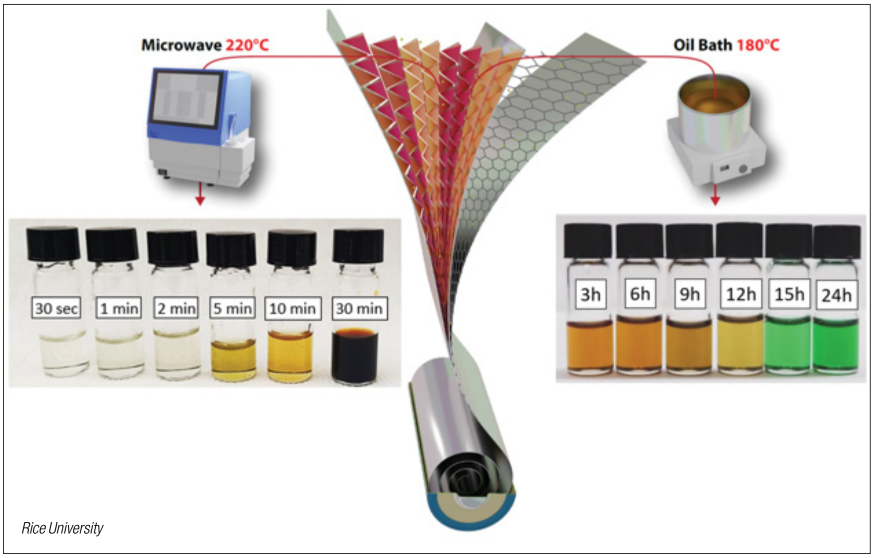

FIGURE 3. Leachate solutions after microwave leaching show yellow or brown shades due to the selective leaching of lithium. The same reaction done in an oil bath yields green colored solutions, since more cobalt and nickel have leached out

ity for spent lithium-ion batteries in Oklahoma. The site will be the first-ever to employ Blue Whale Materials' proprietary technology to process end-of-life batteries (with a range of battery chemistries) and production scrap into a "highly concentrated dry product containing cobalt, nickel and lithium as well as copper and aluminum products." Startup is slated for late 2024.

Last year, American Battery Technology Company (ABTC; Reno, Nev.; www.americanbatterytechnology.com) began operation of a first-of-its-kind, commercial-scale Li-ion battery recycling facility located in the Tahoe-Reno Industrial Center (TRIC) in McCarran, Nev. This commercial facility has the capacity to process over 20,000 m.t. of battery feedstock materials per year when fully ramped, and this first phase of operations will process these battery feedstock materials into recycled products including copper, aluminum, steel, a lithium intermediate and a black mass intermediate material. In July, the company announced that it entered into a milestone purchase agreement with a domestic customer related to recycled black-mass material from its site.

One of the greatest uncertainties in the supply chain will be the handling of end-of-life battery materials and whether a streamlined infrastructure can be established to maximize the recovery of lithium and other critical materials from spent batteries. "We don't know how much of a challenge this is ultimately going to be yet, because we don't have good data, but there are a couple of things that give me hope," says BATT's Gillard. "The automotive manufacturers don't want their batteries to end up at disparate dismantling facilities, so they have a need to close that supply-chain loop for themselves, because there will be a need for that material. They also are offering more modular battery designs that are much easier to take apart, both for repair and recycling. I think it's hard to know exactly what's going to happen with the recycling aspect because there are a few more variables," he notes.

Microwave-based recycling. While the majority of battery-recycling processes are based on hydrometallurgical principles, researchers at Rice University (Houston; www.rice.edu) have developed a new methodology for battery recycling that reportedly provides ultrafast lithium recovery with high selectivity using a combination of microwave reactor technology and an environmentally benign deep-eutectic solvent (DES). "In battery recycling, sometimes less than 5% of lithium is recovered. This is because lithium is usually precipitated last after all other metals, and hence, the solution gradually becomes contaminated, with lithium loss in each step. It was important to devise a process that can quickly and selectively leach lithium," explains Rice postdoctoral fellow Sohini Bhattacharyya. The Rice team has demonstrated, at the laboratory scale, the leaching of a 50% lithium stream in just 30 s. "Just as a microwave heats food much faster than a stovetop, the microwave used here accelerates the leaching process, making this process more commercially viable than other DES-based battery-recycling processes," adds Bhattacharyya.

Since the process follows the same types of procedures (heating, leaching and so on) as typical hydrometallurgical recycling processes, the industrial-grade microwave reactor could easily be inserted into an existing hydrometallurgical recycling setup to help improve lithium recovery.

The DES composition used in this work, a mixture of ethylene glycol (EG) and choline chloride (ChCl), was carefully studied to optimize lithium selectivity. The team's previous work showed that the leaching of transition metals, such as cobalt and nickel, require the involvement of both EG and ChCl, while lithium can be leached with only ChCl (Figure 3). Taking advantage of ChCl's beneficial microwave-absorbance properties, and EG's poor microwave absorbance, only the ChCl is activated in the microwave reactor, rapidly recovering only lithium in this part of the recycling process, and inhibiting the co-leaching of cobalt and nickel. "This demonstrated that tuning DES composition can be instrumental in achieving selectivity towards specific metals in certain conditions," emphasizes Rice doctoral alumna Salma Alhashim.

Finding new pathways for battery recycling will be increasingly important as the U.S. continues to cultivate its own reserves of critical metals. "These materials are, in fact, available in spent lithium-ion batteries, which often end up in landfills. If instead, they are collected and recycled properly, the U.S. can have a more uninterrupted supply of these minerals," says Pulickel Ajayan, the senior researcher on the study and Rice department chair of materials science and nanoengineering. ■

Mary Page Bailey

Holistic Maintenance and Reliability Programs

When updating equipment protection systems in a plant, it often pays to think long term. Today's protection systems can do more than avoid catastrophic failure — they are the foundation of an enterprise-wide predictive maintenance program

Erik Lindhjem
Emerson

IN BRIEF

Chemical process industries (CPI) plants often operate expensive, complex equipment across a number of critical applications, and maintenance teams need to make sure that equipment continues to run to prevent critical processes from shutting down. If that dynamic is not stressful enough, many of those same pieces of equipment can pose dangers. If a compressor operating at 10,000 rpm liberates a blade, the results can be disastrous to personnel, property and reputation.

Fortunately, solutions to prevent catastrophic failure have existed for decades, and plants have been equipping their most critical and dangerous equipment with basic machine-protection devices for a long time. But today, that dynamic is changing. As decades-old equipment is heading into its last rounds of life, and as competitive organizations expand and build out new operations, teams are making changes and updating systems. As they do so, they are considering a new model of maintenance and reliability for critical equipment.

A key element of the coming maintenance and reliability evolution is founded in a boundless automation mindset, as teams are no longer looking to simply protect their equipment and trip it before catastrophic failure occurs. Today's teams know that data give them the power to improve performance, safety and sustainability, so they are looking to collect and move those data seamlessly from intelligent sensors in the field, through the edge, and into analytic and diagnostic systems in the cloud.

Instead of replacing legacy protection systems with the same basic relays that have been used for years, forward-thinking teams are instead using modernization opportunities to build a long-term roadmap to more reliable operation. That roadmap is built on a layered approach, starting with smarter protection equipment that can later be expanded with additional modern machinery-health technologies, ultimately creating a holistic, real-time maintenance program across the enterprise.

FIGURE 1. A unified machinery-health interface makes it easier for any technician to function at their best, regardless of location, helping small teams accomplish more over a larger area

Traditional protection programs

Maintenance and reliability programs are built on protection systems for critical rotating machinery, but traditional protection systems often have significant limitations and fall short of modern needs. Most basic protection systems cannot easily tie back into a plant's distributed control system without complex engineering work. Twenty years ago, when plants had a deep bench of experienced personnel, engineering and maintaining complex, custom connec-

FIGURE 2. When machinery health software collects data from multiple machines and delivers it to a centralized location, like the control room, maintenance and reliability personnel can more easily monitor the health of critical equipment

tions in the face of system updates was a reasonable task.

Today, however, teams are typically leaner and less experienced. But even for teams with the expertise to configure such a connection, when experts move on, the team likely must start from scratch as they try to manage and maintain their custom solutions.

Traditional protection systems also strain lean teams with their need for technicians to travel to the control room or field cabinet to view data. In a single plant, it may be feasible for a technician to regularly travel to the equipment to collect critical data, but this procedure still has users wasting time on low-value tasks. And in a fleet of many plants, traveling to a site could mean someone crossing the continent or the globe if a local site does not have the expertise necessary to analyze data and determine root causes. These extra steps can dramatically increase the time and cost of repair.

If there is no standardization for the protection systems an organization installs across its fleet, technicians can quickly find themselves needing to be experts in dozens of different systems. Plants must also maintain an inventory of hundreds of parts from many manufacturers, further increasing cost and complexity.

Perhaps the most significant problem, however, is that most traditional protection systems are not designed to also support the predictive data necessary for providing advanced warning of developing problems. They may alert maintenance teams that vibration is high, and that protection system will likely — if properly maintained — shut off the machine before catastrophic failure occurs. By the time the system triggers, however, equipment has likely undergone significant damage, and operations have likely suffered from poor performance for some time.

Protection system flaws

As an example, for one global industrial-process manufacturer, a disparate array of legacy protection equipment across its fleet had become a serious problem. The team had hundreds of protection systems across dozens of sites, all from a wide variety of manufacturers. Over time, many of those systems became poorly maintained. Bad wiring, changed relays and loose parts on protection systems occurred at many of the sites.

Trying to maintain a depot of hundreds of parts from different manufacturers had become untenable, and each new technician added to the team had to learn a variety of different systems and techniques to be effective in the field. Reliable maintenance was simply unsustainable, and many pieces of critical equipment were running to failure.

As part of its modernization, the manufacturer chose to standardize across its fleet with a new protection system built for predictive maintenance. The system they installed provided the required powerful protection options, with the flexibility to expand configuration to provide prediction data so the team could better see what was happening on their critical devices.

More importantly for the success of their program, however, was standardization on a single system type across the fleet (Figure 1). The team now has consistency of maintenance, and the company now needs only a single supply center, with a smaller inventory of parts. This new supply system not only reduces the cost of inventory, it also helps the maintenance team more quickly learn which parts they need for any given task.

In addition, the maintenance team only needs to know a single type of protection system, which reduces training requirements and mistakes in the field. Everything looks the same, so it is easier for anyone on staff to perform maintenance work, whether they are working in their home plant or traveling to another site elsewhere in the fleet. Today, it is rare that the team must return to the supply center because they brought the wrong parts or tools to address an issue.

Machinery health visibility

The maintenance and reliability team working for the aforementioned process manufacturer faced two significant problems that helped them determine the next steps in their roadmap. First, critical equipment could only be taken offline at rare times for short periods because the organization needed to continuously manufacture its product. Second, because the team was small, any outages needed to be highly organized, so the maintenance team would not go in "blind."

If technicians were going to work on a piece of equipment, they needed to know exactly what work was required. They also needed to ensure they had parts and the right tools on hand so there would be no

delays in maintenance.

Fortunately, because the organization standardized on a protection system with prediction capabilities, they were able to expand their investment with additional hardware that could be configured to acquire prediction information. Vibration data coming from the protection system, for instance, offered additional value that could help a maintenance team better predict what is happening to the equipment. For example, specific spectrum and waveform data can help differentiate among different common conditions. Those foundational data can be sent to a historian, where analysts can use it to differentiate among rubbing, imbalance, poor lubrication or other conditions.

It is at this stage where the foundation built on powerful, modern protection solutions truly begins to show its value. Once teams have predictive data available for their critical equipment, they can move those data from the historian into fit-for-purpose machinery-health software. Using machinery health software, the team can collect and store data from multiple machines in a single location, where they can use comprehensive analysis tools to assess the health of every piece of equipment quickly and easily (Figure 2).

Machinery health software starts maintenance and reliability teams down the path of continuous condition monitoring. The most effective solutions provide a comprehensive health score for each critical asset in an intuitive format. Technicians can instantly see which machines need attention, and they can drill down further in the software to see the reason for the alert, alongside suggestions for intervention.

More importantly, teams can identify failing equipment well before that failure begins to cause irreparable damage, and long before it trips the protection system. Armed with this rich data, teams can more easily and effectively schedule maintenance outages, limiting disruption to production.

Bringing the plant together

When basic condition monitoring and predictive maintenance is built on a healthy foundation of standardized, powerful predictive systems, it becomes much easier to reach the next step in a maintenance and reliability roadmap, bringing the whole plant together. A powerful machinery-health solution can connect to the plant's other predictive maintenance technologies, such as device and instrument management software, valve management solutions and more to help build the context necessary for better analysis.

Instead of simply looking at discrete data for a single system, teams can now take the vibration data, for example, from their protection and prediction systems and see it in context of operational data. With the many sensors available in the field, teams can loop in temperatures, pressures, flows and more — as well

FIGURE 3. As fans in rotating machinery operate, the blades undergo stress caused by vibration and resonance. Machinery health solutions can help reliability teams identify excessive vibration before it causes damage

as information about changes in the process — to see what is really happening in the plant.

For example, consider a team at a chemical facility that recently went into a short-window outage trying to determine the cause of high thrust measurements on a centrifugal compressor. The team used much of its outage time examining equipment, and eventually discovered that a control valve was no longer functioning as intended. That valve failure created a compressor surge condition that was seen in the high axial-thrust measurements and high radial vibration caused by a mechanical rub.

By the time the team discovered the cause, the compressor was already damaged. As a result, during the outage the team not only had to tear down and rebuild the valve, but they also had to perform significant compressor repairs.

The protection system functioned as intended by keeping the compressor from catastrophic failure, but this was not enough to prevent equipment damage. If the team had been using a machinery health solution tied into both the vibration readings from the compressor and the signatures from the valve, they would have had the contextual data necessary to identify exactly what was happening long before they started shutting equipment down, allowing them to save time and money (Figure 3).

Today's industrial equipment is relatively stable, and it is unlikely that a part like a fluid film bearing will fail on its own. As a result, it has become far more important to see a holistic picture of how every asset in the plant contributes to the health of critical equipment. Moreover, such visibility also reduces or eliminates recurring problems. If a bad valve or bad lubrication-oil system is causing bearings to fail, those bearings will keep failing until the root cause is remedied. Without a holistic view of plant health, such problems are very hard to find and fix.

Better analytics

As organizations improve their analysis on the plant level, they will ultimately want to move their data up to the enterprise level, where cross-functional teams can use it to innovate and improve performance. When a plant's maintenance and reliability solution is built on a connected, predictive foundation — from protection to machinery health to balance-of-plant monitoring — high-level teams can more easily make critical business decisions.

For example, if a team can not only discern that a problem exists, but also identify the steps to remediation and the amount of time the system can run without creating additional costly damage to critical equipment, the organization can better decide whether to shut equipment down immediately for maintenance or wait until the next scheduled outage.

Even more significantly, however, those same data can tie into enterprise-level software to help connect operations and maintenance, and empower them with advanced analytics, automated workflows, decision support and more. Armed with such tools, teams can more easily and effectively make collaborative decisions, such as whether to extend intervals between maintenance to drive increased production to meet an emerging need.

Think lifecycle

As the time comes to update protection systems around the plant and across the enterprise, it often pays to think long term. While like-for-like system swaps will help ensure critical protections are in place, they can also cause additional headaches and are often not designed with a long-term holistic maintenance program in mind.

To meet the dynamic needs of today's marketplace, organizations are better served by implementing a foundation of advanced protection systems, designed to be expanded into predictive maintenance, and, ultimately, an enterprise-wide holistic reliability and maintenance system. Such a solution delivers improved maintenance today, while preparing the team to continue improvement over the lifecycle of their equipment. ■

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All figures courtesy of Emerson

Author

Erik Lindhjem serves as vice president and general manager of Emerson's Reliability Solutions business (pss.marcom.emerson.com). In this role since June 2021, he has been focused on driving digital transformation through plant asset management of automation assets and machinery that enables clients to reach top-quartile performance. Lindhjem joined Emerson as vice president of Reliability Solutions and Consulting, Asia Pacific in August of 2018 and was based in Singapore. He holds a B.S. degree in mechanical engineering from the University of Virginia and an M.S. degree in business administration from Wake Forest University.

Averting Disaster: Delivering Value from Sensors and Alarms

A strategic approach to asset and alarm management will reduce alarm fatigue, mitigate risk and support more effective response

Tacoma Zach
MentorAPM

IN BRIEF

ASSET OVERSIGHT

IDENTIFY CRITICAL ASSETS

PUT THE RIGHT PROCEDURES IN PLACE

ALARMS AND ASSET MANAGEMENT

ASSET AND ALARM INVESTMENT PLAN

ALARM RATIONALIZATION

On April 20, 2010, an explosion on the Deepwater Horizon offshore drilling rig (owned by Transocean and operated by BP) killed 11 crewmen. The resulting fire was inextinguishable, and the rig sank to the ocean floor two days later, leading to the largest marine oil spill in history.

During the U.S. Coast Guard and Bureau of Energy Management joint investigation into the explosion [*1*, *2*], Mike Williams, chief electronics technician for Transocean, was asked: "Did you at any time hear any alarm that would indicate a general muster?" His response: "Never."

As chief electronics technician, Williams was responsible for "maintaining the fire and gas systems and any and all electronic signaling devices throughout the rig." During his testimony, it was revealed that the alarm system had been "inhibited," which is an alarm condition where the sensors are active but will not trigger an alarm. It was also revealed that the alarm system had been bypassed for longer than a year because "they did not want people to wake up at 3:00 in the morning due to false alarms [*3*]." This unfortunate decision resulted in the deaths of 11 people and injuries to 17 others.

Asset oversight

Sensors, alerts and alarms are critical to the safety, reliability and operations of manufacturing plants and only deliver value when they are trusted and when the right action is taken at the right time, as was so tragically demonstrated on the Deepwater Horizon. Organizations that adopt a strategic asset management approach in their operations can apply that framework to reduce alarm fatigue, mitigate risk and support effective response.

In the past, only the most critical systems were monitored because sensors had to be hardwired from the asset to the control room. At $100 per foot for wiring, it was economically prohibitive to monitor all essential assets. With the advent of inexpensive wireless sensors and the industrial internet of things (IIoT) technologies, manufacturers are looking to reduce safety risks and improve operations by implementing online condition-based monitoring throughout their facilities.

As with most digital transformation initiatives, manufacturers struggle with where to start. Their struggle is understandable. According to McKinsey, more than 70% of all digital transformation initiatives do not yield the intended results [*4*]. Industry best practices suggest starting small

FIGURE 1. Control system design may often have numerous alarms incorporated that can be triggered simultaneously, making it difficult to ascertain the root cause of the instigating incident

and identifying projects that will yield positive results. Historically, this has meant installing sensors on assets that mitigate risk and improve reliability. One obvious example is to put sensors on equipment in hazardous environments that pose a substantial safety risk, like cooling tower fans.

As noted above, sensors and alarms are only valuable when personnel acknowledge the alerts and take the proper corrective action. On Deepwater Horizon, Williams acknowledged that he became "immune" to the constant barrage of alarms. This is a common problem in many distributed control system (DCS) control rooms (Figure 1). During the design and implementation of DCS systems, engineers will incorporate alerts and alarms for various equipment based on multiple variables. This approach often results in multiple alarms being triggered without an indication of the root cause of the most critical alarm. To minimize "nuisance" alarms, system designers can perform alarm rationalization to create an effective alarm system that evaluates each alarm to determine its relevance, importance and impact on the process, as well as the operator's effectiveness.

Identify critical assets

Underlying the alarm rationalization process is the assumption that all assets have been prioritized with regard to importance, criticality and impact to the process. This is typically done through criticality analysis, but unfortunately, many companies assume they already know the critical systems and assets and find no need to perform a criticality analysis. In many cases, they do know about 80% of what is critical, but it's the other 20% that will end up causing the most problems.

In the case of Deepwater Horizon, the internal BP investigation found [*1*, *2*] that "the design of the gas detection system was apparently based on a single online combustible-gas detector (CGD) at each location. Such a system lacks the redundancy levels associated with a high-reliability design. If a single CGD did not work, was inhibited, or in some other way was out of commission, the protective functions provided by that device would have been lost."

FIGURE 2. A holistic asset-management platform can incorporate workflow procedures alongside alarm and diagnostic data to help ensure that proper corrective actions are completes

The result of the lack of CGD redundancy is one of the key findings of the investigation: "The fire and gas system did not prevent hydrocarbon ignition." A properly performed criticality analysis should catch key factors like the lack of necessary redundancy in an automated safety system. The great benefit of performing a criticality analysis is that it will indicate the right things to do, in the right order, for the right reasons, and to get the right results that align with the plant's objectives.

From a criticality analysis, reliability and maintenance personnel can prioritize which assets need online monitoring through sensors. Furthermore, plant managers will have confidence that not only are sensors being installed on the right assets, but also that their digital transformation projects will be successful.

Put the right procedures in place

In the excitement of "going digital," many manufacturers do not take the time to make the necessary changes in their current work procedures to take advantage of new technologies. One of the reasons for conducting a pilot or proof of concept is to uncover areas that need change management.

For example, consider the experience of conducting an online condition-monitoring pilot project using wireless vibration sensors on rotating equipment. The online condition-monitoring-solution vendor's systems engineers were monitoring the equipment along with the client's maintenance department. One of the engineers had noticed an alarm had triggered and yet no acknowledgement of the alarm had taken place within the system. When the engineer called the client, the maintenance person confirmed they had received the alarm, but they had no procedures on how to respond. The problem was rectified by also notifying the reliability engineer and developing and updating the work processes. Any deployment of new technology will warrant a review of affected work processes and additional changes.

Alarms and asset management

An alarm is only as good as the responding action. A sensor can quickly identify a fault condition and provide insight to operators of potential problems months before a failure. However, it is imperative that once an alarm is received, the work procedures for corrective action are readily available to the maintenance crews in the field. Work-order management systems that are integrated into an end-to-end asset-management system can capture the task procedures and display in real-time the workflow progress to assure supervisors that the corrective actions have been properly followed and completed (Figure 2).

More importantly, an end-to-end asset-lifecycle management system, also known as an enterprise asset-management solution, incorporates the correct asset interven-

tions and strategies that are foundational to an alarm rationalization implementation. As plant operators and maintenance crews start to retire and leave the workforce, asset management systems can capture their institutional knowledge to assure future plant personnel deploy the correct intervention for the various asset classes when alarms are received.

The advent of inexpensive wireless sensors has resulted in a proliferation of online condition-based monitoring systems. Real-time alerts from these sensors allow plant personnel to become more proactive to potential problem situations. Furthermore, precious maintenance resources are more effectively deployed to assets based on their condition from sensor data, which reduces unnecessary, time-based preventative maintenance.

Asset and alarm investment plan

Incorporating alarm management into an asset management system can have a major impact in moving manufacturers from a reactive position to a proactive or predictive stance in their maintenance and reliability strategies. One such area is asset lifespan. As enterprise asset management systems intake more real-time data, manufacturers can pinpoint assets that are approaching their end of life far in advance. Building on the criticality analysis, companies can utilize an asset investment planning (AIP) application to rank the capital improvement projects that will have the greatest impact on reducing risk and improving operational excellence.

As a case in point, a large refinery had a capital improvement plan for a boiler-feed pretreatment system. They had conducted several design, HAZOP and construction reviews. Given the importance of the system, they decided to conduct a criticality analysis to support their planned improvements. After 18 years of operation, they did not expect to find any unidentified issues and were therefore surprised by previously unidentified risks that were uncovered during the analysis. One such risk was a small brine system that would have brought the facility to a full shutdown in less than 8 hours if it failed. The four-week lead time to replace this system was an unknown and unacceptable risk. The capital plan was quickly adjusted to include a relatively small $10,000 asset investment that averted a potential $25 million risk.

Alarm rationalization

Operators can experience a dramatic reduction of risk by considering alarm rationalization as an asset management strategy. The advent of wireless sensors and the internet have launched a proliferation of technologies for alarm management within the industrial marketplace, making it increasingly easier to create a cacophony of alarms. The explosion on Deepwater Horizon demonstrated that more is not necessarily better, and it can, in fact, be deadly. Now more than ever, it is important that all alarms are relevant and meaningful with clear procedures for response. By supporting alarm rationalization with an asset management strategy through an asset lifecycle-management platform, industrial manufacturers can dramatically improve their operations, reduce their overall risk and optimize their capital investments. ■

Edited by Mary Page Bailey

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Author

Tacoma Zach is co-founder and CEO of MentorAPM asset-performance and work-management software solutions (2416 E Goldenrod St., Phoenix, AZ. 85048; Phone: 602-492-6212). Having spent most of his career in the operation and management of both municipal and industrial water/wastewater operations, Zach is an expert on the application of asset management best practices, risk management and ISO 55000 standards to w/www utilities. He is the author of Criticality Analysis Made Simple and speaks frequently at leading asset-management conferences. He holds B.S.Ch.E. and M.S.Ch.E. degrees from the University of Toronto and is a licensed Professional Engineer in Ontario, Canada.

Pad-Reinforced Nozzles in Pressure Vessels

This article examines the application of reinforcing pads for strengthening nozzles in pressure vessels and the impact of the geometric gap that exists between the pad and the vessel shell

Walther Stikvoort
Independent Consultant

Many common forms of nozzle connections in technological applications are subjected to internal pressure and external loads. Pressure-vessel design engineers are often confronted with various considerations that they have to understand with regard to the nozzle design. Practice shows that the traditional design codes do not always provide adequate solutions for specific cases and do not sufficiently address relevant points that are important to ensure structural integrity, including the reinforcing pads that are often used for strengthening nozzles in pressure vessels that are subjected to internal pressure and piping reactions (Figure 1). Moreover, it cannot be confirmed that the resistance to gross plastic deformation (GPD) of a pad-reinforced shell is exactly equal to the resistance of a single-wall shell whose thickness is equal to the sum of the pad and shell. GPD under static loading is a fundamental failure mode that must be considered in the design by analysis (DBA) method for pressure vessels. In elastic DBA, GPD is prevented by limiting the primary stress in the vessel. In inelastic DBA, GPD is prevented by limiting the load applied to the vessel, restricting it to a fraction of the notional ductile collapse load of the vessel.

There are a number of parameters that are important to consider for pad-reinforced (repad) nozzles. This article discusses several aspects that design engineers must take seriously in order to ultimately achieve a prudent and safe nozzle design.

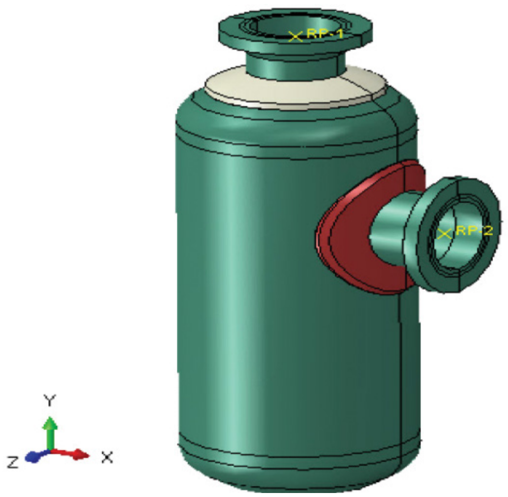

FIGURE 1. A typical pressure vessel with pad-reinforced nozzles is shown

Characteristics of repads

Perfect contact between the vessel shell and the pad cannot be achieved for several reasons. First, this contact adversely affects the stress distribution in the vicinity of the nozzle intersection and results in a lower internal pressure capacity compared to a fully integral connection without a gap.

However, in most design codes, the gap between the pad and the shell, which prevents full contact between the two elements, is ignored, and the thickness of the pad plus the shell is considered integral, which raises some questions. Ref. 1 provides additional insight into the effect of a geometric gap between a cylindrical or spherical shell and a reinforcement pad.

The statement that a gap may (but not always) exist between the pad and the main vessel shell is in fact correct. This is due to the skill level of the sheet-metal worker who rolls and forms the material to make the pad.

All the nozzle-reinforcing calculations assume the pad, weld, shell and nozzle form an integrated assembly — this also accounts for internal pressure and for external piping loads.

However, there have been some occasional instances where poor fit does indeed play a part, especially if there is thermal expansion and no weep hole. A weep hole is a small hole drilled in the pad to allow any gap moisture to be released. If there is no weep hole, then the gas in the inner space can expand. In such cases, it is possible for the pad to "blow off" and crack the welds or the vessel itself.

Discussion

The region of intersection of a nozzle with a pressure vessel is generally one in which high stresses exist. Consequently, the designer of a pressure vessel must have means for accurately predicting the magnitudes and locations of maximum stresses in these regions.

According to Welding Research Council (WRC) Bulletins 107/297/368, which are frequently used in performing nozzle load analyses, if the width of the shell reinforcement (pad plate) is equal to or greater than the below expres-

TABLE 1. EQUATIONS AND RESULTING CALCULATIONS (FOR EXAMPLE)

	Equations	Elaborations	Results
(1)	$t_e=(T_v^{2.5} + t_p^{2.5})^{0.4}$	$t_e=(20^{2.5})+12^{2.5})^{0.4}$	22.067 mm
(2)	$0.5\, t_s \leq t_p \leq 1.5 t_s$	$0.5 \times 20 \leq 12 \leq 1.5 \times 20$	Satisfied
(3)	$W \geq r\,[(\,t/t_s\,)^{0.75} - 1]$	$W \geq 203.2\,[(32/20\,)^{0.75} - 1]$	80 mm < 85.877 mm
(4)	$W \geq 0.611\,(R.t)^{0.5}$	$W \geq 0.611\,(800.32\,)^{0.5}$	80 mm < 97.76 mm
(5)	$t_r = 2.678 W^2/R$	$t_r = 2.678 \times 80^2 / 800$	21.424 mm
(6)	$t_r = t_s\,[(r + W)/(r)]^{4/3}$	$t_r = 20\,[(203.2 + 80)/(203.2)]^{4/3}$	31.132 mm

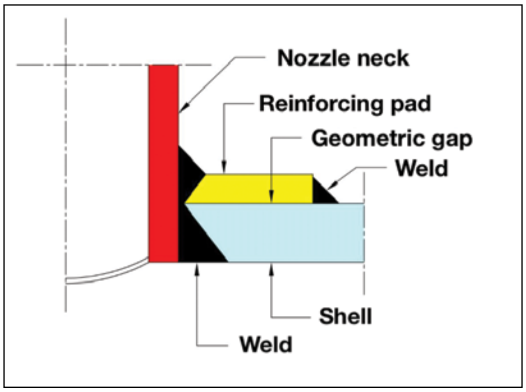

FIGURE 2. This illustration of a pad-reinforced nozzle shows the presence of a geometric gap

sion, where R is the shell radius, T is the vessel thickness and d is the nozzle diameter:

$$1.65(RT)^{0.5} \text{ or } d/2,$$

then it can be assumed that the thickness of the reinforced portion of the shell would determine the state of stress at the nozzle-to-shell junction. In such a case, the T value to be used in equations for calculating stresses should be T (the shell thickness plus the pad plate thickness). It is generally assumed that discontinuity stresses due to the penetration have been reduced to negligible levels at a distance of $1.65(RT)^{0.5}$ or $d/2$, whichever is greater, from the nozzle neck. Stress attenuation is the key here.

Ref. 2 recommends that in finite element analysis (FEA) studies, the analysis not be based on the total pad plus vessel thickness, but on a thinner effective pad thickness that represents the bending stiffness of a separate pad and vessel shell, but is conservative for local membrane stress. When the local membrane stresses govern the maximum loads, an additional FEA evaluation should be performed using full thickness. The effective vessel shell plus pad combined thickness (t_e) is given by the following equation, where: T_v represents vessel thickness and t_p is the reinforcing pad thickness:

$$t_e = (T_v^{2.5} + t_p^{2.5})^{0.4}$$

Ref. 3 states that if the reinforcing pad of adequate width has been applied, the total shell thickness can be assumed as the corroded shell thickness plus the pad thickness. Criteria for adequate pad width is as follows:

$$0.5\, t_s \leq t_p \leq 1.5\, t_s$$

$$W \geq r[(t/t_s)^{0.75} - 1]$$

$$W \geq 0.611\, (Rt)^{0.5}$$

Where:
W = width of reinforcing pad (mm)
R = external radius of shell (mm)
r = external radius of nozzle (mm)
t_s = thickness of shell (mm)
t_p = thickness of reinforcing pad (mm)
t = combined thickness of shell and pad (mm)

Note that if the pad is not wide enough, then t should be replaced by a reference thickness t_r. To determine t_r, use the lesser of the following:

$$t_r = 2.678\, W^2/R$$
$$t_r = t_s[(r + W)/(r)]^{4/3}$$

Example

Consider an example of a cylindrical shell with an outside diameter of 1,600 mm and a wall thickness of 20 mm. A 16-in. NPS (NB 400) flush nozzle is fitted in the cylindrical shell, which is provided with a rein-

forcing pad with a thickness of 12 mm and a width of 80 mm. Table 1 outlines the relevant equations and results for this sample calculation.

Since the requirements of Equations (2) and (3) are not met, it is not permitted to take the total thickness of the shell plus reinforcing pad into account. This means that a substitute or equivalent thickness of 21.424 mm must be applied. It should be noted that this result differs little from the equivalent thickness calculated with Equation (1). The difference is approximately 3%.

Refs. 4 and 5, which use the so-called pressure-area method for the nozzle compensation calculation, which is common in European codes, introduces a reinforcement efficiency factor *k* of 0.75 for the reinforcement pad. This means that the cross-sectional area of the pad within its defined boundaries should be multiplied by this factor in order to compensate for the gap. It should be noted that the implementation of this *k* factor applies specifically to the design code mentioned here (from the Netherlands) and has not been adopted by the other European codes like EN 13445 (EU), PD 5500 (U.K.), AD 2000 (D) and CODAP (F).

Note that ASME BPVC Section VIII-Division 1 — paragraph UG-37 (g);(h), however, indicates that the area A5 being the area of the reinforcing pad should be multiplied by 0.75 if some provisions are not met.

Findings

It can be concluded that views regarding the contact problem between the repad and shell — and the way in which this situation should be anticipated — differ considerably. A more in-depth investigation into this by means of a numerical analysis method (such as FEA) under different loading regimes (internal pressure and imposed pipe loads) and simulating the gap in combination with varying contact surfaces between the repad and shell could lead to workable solutions. However, modeling of pad-reinforced nozzles is quite complex due to the possible gap between the pad and shell, or to some extent, the lack of contact between both elements [*6*]. Some computer programs are available to design engineers that offer solutions for this. However, it is preferable to develop a specific analytical approach. The vessel design engineer must be aware that ignoring the effect of the gap can result in a non-conservative prediction of the load capacity. ■

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Acknowledgement

All figures provided by author unless otherwise noted

Author

Walther Stikvoort (Email: stikvoort@ziggo.nl) is a renowned authority in the field of mechanical and structural integrity of static pressure equipment. He has more than 50 years of experience in pressure vessel and piping design and has developed numerous technical standards and practices to improve the asset integrity of leading operating companies. He is the author of numerous peer-reviewed international journal articles in the field of mechanical and structural integrity. During his career, he was regularly active in developing and teaching courses and training to mechanical engineers in his area of expertise and he was a member of various expertise committees. He is currently active as a consultant on static pressure equipment integrity serving the engineering community on request.

Further reading

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Separating Hazardous Materials with Decanter-Centrifuge Systems

Sealed and gas-purged decanter-centrifuge systems can be effective for separating Class I, Div. 1-, or Class I, Div. 2-rated solvents and hazardous materials

Lee Betkowski
Flottweg Separation Technology Inc.

Traditionally, solids and liquids have been separated by filtration or with press equipment. While these methods are effective, modern automated separation technologies, such as decanter-centrifuge systems, are excellent alternatives for faster separation and a more efficient approach.

When processing flammable, hazardous or otherwise dangerous material that can adversely impact the environment, it is important to prevent it from escaping the process stream. It is equally important to prevent atmospheric oxygen from entering the centrifuge system and creating an unsafe environment that can potentially cause an explosion or to prevent toxic materials/vapors from leaving the machine to protect operators in the area. A purged centrifuge is an inert-gas-blanketed system that forms a "technically tight" seal between the interior of the centrifuge and the environment.

Sealed centrifuges can be used safely and effectively while avoiding these types of situations. In some cases, it is important to minimize exposure to atmospheric oxygen to prevent explosions, but it may also be necessary to prevent hazardous material from escaping during processing. Some applications in which sealed, inert-gas-blanketed centrifuges are used include the following:

- Oil and industrial sludges
- Tar cleaning
- Drilling mud and emulsion processing
- Industrial and chemical waste processing
- Solvent extraction processes
- Chemical processes using flammable solvents
- Fermentation applications using flammable media

This article explains how technically sealed decanter centrifuges, blanketed with inert gas, improve efficiency and safety in dewatering and separating processes of combustible or hazardous materials.

FIGURE 1. Continuous-flow purged centrifuge systems are "technically tight," which is different from being hermetically sealed

Flammable solvent	LOC [Vol%]
Methanol	8.1
Ethanol	8.5
Isopropanol	8.7
Hexane	9.3
Xylene	9.7

FIGURE 2. Minimum oxygen concentration is the limiting oxygen concentration (LOC) below which combustion is not possible

Purging principles

There are three general approaches for inert-gas purging of process equipment, as described below.

Overpressure. This method can only be used with equipment that is designed for over-pressure. The air inside is removed by continually adding and releasing high-pressure inert gas into the sealed equipment. This step is repeated until the oxygen concentration inside the equipment measures at the desired level. Then, while the equipment is running, the internal pressure is held at a higher differential pressure than the atmospheric pressure (though this operating pressure is still much lower than that used during inertization).

Vacuum. All air inside the equipment is removed by evacuating the equipment, at which time, the vacuum is broken by flushing with inert gas. Depending on the equipment, this procedure must be repeated. Equipment designed for holding vacuum is required for this process.

Continuous-flow purging. This method is used for plants, machines, containers and equipment that are designed for high overpres-

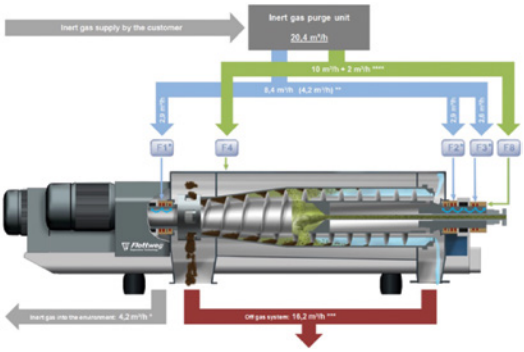

FIGURE 3. The diagram shows the pre-purge flow of a decanter centrifuge system

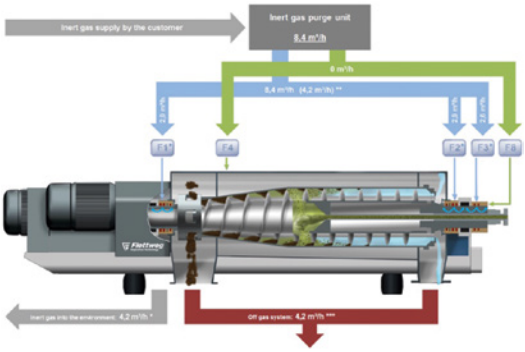

FIGURE 4. The blanketing purge flow is shown here for the same type of decanter centrifuge

sure or vacuum. It works by feeding inert gas into the equipment at one point in the closed system and simultaneously releasing it at another point a distance away from the infeed. There is typically a pre-purge sequence at higher flowrates, and a blanketing sequence during normal operation of the equipment.

The centrifuge systems discussed in this article use a version of the third method (continuous-flow purging). They are built to Class I, Div. 1 or Class I, Div 2 (U.S. National Electrical Code (NEC) 500) standards. Continuous-flow purged centrifuges can also be designed for Canadian NEC 505 and are currently available for European ATEX Directive (2014/34/EU). Design requirements are subject to different standards in different countries. The focus here is on NEC 500 classifications.

It is also important to note that continuous-flow purged centrifuge systems are "technically tight," which is different from being hermetically sealed. A hermetically sealed piece of equipment provides a zero-leakage tolerance, while the "tightness" in a technically tight system cannot be permanently ensured, due to its design, function and so on (Figure 1).

Two protection concepts

There are two protection philosophies used for applications that require continuous-flow purging. Both can be customized to specific materials and settings.

Combustible materials. When processing flammable or potentially explosive materials, the goal is to avoid fire or explosion. Because any spark can cause combustion when oxygen is present, an inert atmosphere must be created and maintained inside the centrifuge. This is accomplished by removing oxygen from inside the centrifuge during startup and maintaining an inert atmosphere during operation. Examples of combustible media include alcohols (methanol, ethanol, isopropanol and so on) and solvents (hexane, heptane, octane, acetone, toluene and so on).

Hazardous materials. When processing hazardous materials, the goal is to protect operators as well as the surrounding environment by preventing materials from escaping the centrifuge.

Internal atmosphere for safety

Combustion requires oxygen because it results from a chemical reaction between the combustible material and air. The goal of a sealed centrifuge system is to achieve and maintain the desired limiting oxygen concentration (LOC) inside the decanter system. This is also known as the minimum oxygen concentration (MOC) and is defined as the limiting concentration of oxygen below which combustion is not possible. It is expressed in units of volume percent of oxygen (Figure 2). LOC varies with pressure and temperature as well as the type of inert gas used [*1*].

Steps for low-O_2 atmosphere

Creating an internal atmosphere that meets the desired LOC requires two steps: the pre-purge and inert gas blanketing (Figures 3 and 4).

The pre-purge step occurs before the machine is started. The interior of the centrifuge is flushed with inert gas (such as nitrogen, Figure 5) to displace the air/oxygen. This is accomplished by filling the housing until the pressure exceeds 0.290 psi and continues until the flow of inert gas has replaced the interior volume of the centrifuge multiple times over or until an oxygen sensor indicates a non-hazardous concentration of oxygen, or both. The amount of time this takes varies according to the decanter size, but is usually less than 30 min.

After purging, the inside of the centrifuge is "blanketed" with the inert gas, forming a technically tight seal. In this step, the system is held at a slight overpressure. The unit's control system also maintains a higher pressure at the seals on both ends of the decanter. This results in a constant differential pressure to the atmosphere, which prevents the outside atmosphere from penetrating the inside.

Importantly, the inert-gas supply system must be equipped with sufficient redundancy to guarantee the continuous availability of inert gas during centrifuge downtime, in case of any failure.

Blanketing system operation

A standard mathematical formula is used to calculate the amount of gas required to displace the volume of the decanter. Once an adequate volume has been displaced and safe operating conditions are reached, the system is considered to be in an

Why Use Nitrogen?

Nitrogen is commonly chosen for inert gas blanketing applications because it is nonreactive, readily available, and generally inexpensive. These are the requirements for using nitrogen with a sealed centrifuge system:

- Inlet pressure: 4–6 bar (g)
- Purity level: min. 99 vol % N_2 (max. 1 vol % O_2)
- Filtration degree: 5 µm
- Inert gas temperature: 0–60°C
- Moisture content from the inert gas:
  - pressure dew point ≤ 3°C at temperatures above 3°C
  - pressure dew point ≤ -20°C at temperatures below 3°C

FIGURE 5. Nitrogen is a common choice for inert-gas blanketing applications. The requirements for nitrogen are shown here

operational state (that is, inert). During operation, the differential pressures must be maintained to ensure safety. This is done with an overpressure sealing and monitoring system consisting of approximately five flowmeters, two flow switches and eight pressure sensors (Figure 6).

Specialized mechanical shaft seals prevent the surrounding atmosphere from penetrating into the interior of the centrifuge. They also prevent gases from escaping the inside as long as steady overpressure is maintained.

A seal consisting of several sealing rings separates the interior of the centrifuge from the outside atmosphere. By using multiple sealing elements, including additional seals on shaft feedthroughs and housings, a technically tight seal is created. This also reduces the amount of inert gas used, since the seals help ensure minimal loss throughout the system.

Inert gas is fed between the sealing rings. It flows through narrow seal openings, both into the interior and

ZONES AND DIVISIONS FOR AREA CLASSIFICATION

Hazardous areas are classified into zones to aid in selecting and designing the appropriate mechanical and electrical equipment used in that area. Zone 1 refers to an area in which an explosive atmosphere consisting of a mixture of air and dangerous substances in the form of gas, vapor or mist is likely to occur intermittently during normal operation.

In a Class I, Div. 1 location:

- Ignitable concentrations of flammable gases or vapors can exist under normal operating conditions, or
- Ignitable concentrations of such gases or vapors may exist frequently because of repair or maintenance operations or because of leakage, or
- Breakdown of the equipment or a process failure may release ignitable concentrations of flammable gases or vapors and may also cause simultaneous failure of electrical equipment in such a way as to directly cause the electrical equipment to become a source of ignition.

In a Class I, Div. 2 location:

- This zone is defined as onee in which volatile flammable gases, flammable liquid — produced vapors, or combustible liquid — produced vapors are handled, processed, or used, but in which the liquids, vapors, or gases will normally be confned within closed containers or closed systems from which they can escape only in case of accidental rupture or breakdown of such containers or systems or in case of abnormal operation of equipment, or
- One in which ignitible concentrations of flammable gases, flammable liquid – produced vapors, or combustible liquid – produced vapors are normally prevented by positive mechanical ventilation and which might become hazardous through failure or abnormal operation of the ventilating equipment, or
- One that is adjacent to a Class I, Division 1 location, and to which ignitible concentrations of flammable gases, flammable liquid – produced vapors, or combustible liquid – produced vapors above their flash points might occasionally be communicated unless such communication is prevented by adequate positive-pressure ventilation from a source of clean air and effective safeguards against ventilation failure are provided.

(Source: NFPA National Electric Code, Articles 500 – 504 [Divisions])

FIGURE 6. Differential pressures are maintained with an overpressure sealing and monitoring system

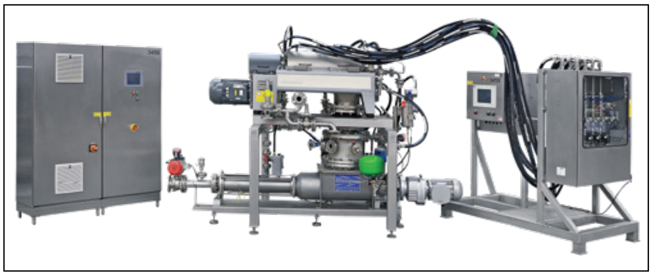

FIGURE 7. Internal oxygen concentrations can be managed in two ways: with oxygen sensors and with a continuous-flow purge system

FIGURE 8. Routine maintenance and testing points include threaded connections, manometers, meters, flowmeters and hoses

to the atmosphere. This is only possible if the pressure at the seal face is higher than the pressure in the decanter housing (>0.290 psi) and the outside atmosphere. The differential pressure (0.725–1.45 psi) between the housing and the gas feed point is monitored and controlled. Seals are pressurized to keeping oxygen from entering the system.

Managing O_2 concentrations

Internal oxygen concentrations can be managed in two ways: with oxygen sensors and with a continuous-flow purge system (Figure 7). Oxygen sensors monitor the oxygen concentration inside the centrifuge housing. An advantage to using these sensors is the continuous monitoring they offer, especially if the differential pressure between the internal housing and the atmosphere is less than 0.290 psi.

Oxygen sensors provide direct information about the risk factor of the oxygen concentration. In contrast, a continuous-flow purge system is indirect and relies on pressure-sensor setpoints. As long as the overpressure on the seals or housing is maintained, no oxygen from the environment can penetrate the system and the oxygen concentration is held below the critical level. This is done with flowmeters and pressure gages.

However, since continuous-flow systems rely on pressure sensor setpoints, applications using lower differential pressures may be susceptible to erroneous system shutdowns. In these cases, oxygen sensors may provide more sensitive monitoring of this danger zone.

Both methods ensure safe operation if they are properly planned, installed and maintained. Unfortunately, the weak point with an oxygen sensor is the sensor itself, which requires programmable logic controller (PLC) programming, calibration, periodic replacement and maintenance. Overall, continuous-flow purging offers better net positive usability when compared with oxygen monitoring.

Safe and efficient separation

Using an inert gas to form a tightly sealed or purged centrifuge is an excellent solution for a variety of applications, including explosion protection in the chemical, pharmaceutical, petrochemical or biodiesel industries; and environmental protection from hazardous materials, such as those generated during tar cleaning.

Once commissioned, a leak-tightness test schedule should be created and performed regularly to verify the tightness of the purge unit. Routine maintenance and testing points include threaded connections, manometers, meters, flowmeters and hoses. Supply lines also must be checked at regular intervals for damage, clogs and tightness (Figure 8).

Centrifuges are extremely efficient, with higher throughput than filters or presses and they operate continuously, instead of in batches as with filters or presses. It is possible to separate two phases, three phases or a very light emulsion with a purged centrifuge. Many systems are also fully automated, which reduces operator involvement overseeing the machine. ■

Edited by Scott Jenkins

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Acknowlegements

All images in this article appear courtesy of Flottweg Separation Technology Inc.

Author

Lee Betkowski has been the biotech, chemical, pharmaceutical (BCP) industry manager for Flottweg Separation Technology Inc. (10700 Toebben Drive, Independence, KY 41051; Email: lbetkowski@flottweg.net) for 13 years. Prior to joining Flottweg, he worked for Krauss Maffei (pusher, peeler centrifuges, dryers), Rosenmund (filter dryers) and De Dietrich (glass-lined steel equipment). Betkowski has nearly 30 years of experience in the biotechnology, chemical and pharmaceutical industries, working predominantly with centrifuge products and technology.

Controlling Process Variables for Solid-State Battery Performance

Optimizing electrical conductivity in next-generation batteries depends on carefully controlling process variables, such as mixing, particle-size distribution and temperature. Presented here is a discussion of how these process variables affect battery performance

Janina Rhea A. Lazo-Cruz and Daniel Shafer, B&P Littleford

In the evolving landscape of energy storage technologies, solid-state batteries (SSBs) have emerged as promising alternatives to traditional lithium-ion (Li-ion) batteries. By employing solid electrolyte materials, SSBs have several potential advantages over traditional Li-ion batteries, which use electrolytes made of liquid solutions. SSBs are capable of higher energy densities than traditional Li-ion batteries, and are not susceptible to the formation of lithium dendrites, which can pose safety risks. In addition to energy density and safety advantages, the substitution of a solid electrolyte material also removes some restrictions in the battery-cell architecture that are present with traditional Li-ion batteries.

In the manufacturing process for electrode materials for both SSBs and traditional Li-ion batteries, slurries of active materials, conductive agents, binders and solvents must be precisely formulated and processed to achieve optimal electrode properties. The electrical conductivity of the battery slurries plays a vital role in dictating the performance of these batteries. Moreover, the batteries' performance and longevity are intrinsically linked to the structure and properties of their electrodes, which are primarily determined during the manufacturing process.

In order to achieve optimal battery performance, processors need to know how various process variables affect the electrical conductivity of battery slurries. Among the various stages of electrode fabrication, the slurry mixing process is particularly critical. This article discusses the process variables related to the mixing process, and how they can affect the particle size and distribution of the electrode components.

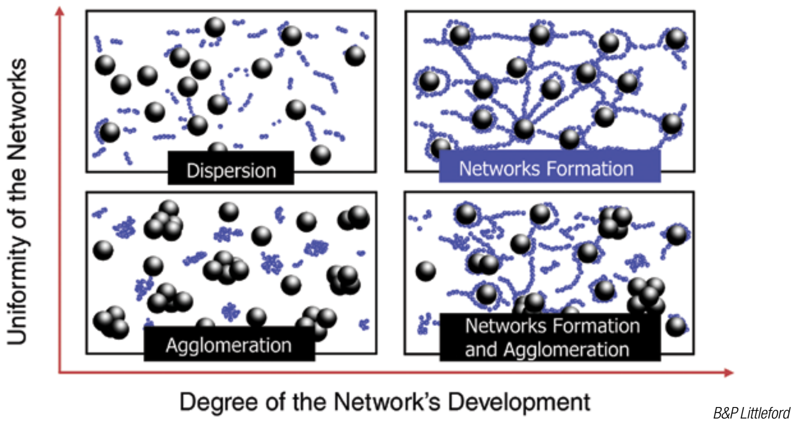

FIGURE 1. Mixing plays an important role in determining ultimate electrode properties. Insufficient mixing can result in uneven distribution of active materials, but overly intense mixing risks degradation of sensitive materials

Mixing time

The duration of the mixing process significantly affects slurry homogeneity. Insufficient mixing time can lead to particle agglomeration, resulting in uneven distribution of active materials and conductive agents. This unevenness can hinder both ionic and electronic conductivity. On the other hand, excessive mixing can introduce thermal degradation of sensitive components and can lead to the degradation of the conductive network, increasing the composite volume resistivity and reducing the battery's performance (Figure 1). Utilizing a continuous mixing system, such as a twin-screw extruder, allows for precise control over mixing time, ensuring uniform dispersion without damaging the materials.

Mixing speed

The speed of the mixer is crucial in applying appropriate shear forces. Higher speeds can enhance dispersion and break down agglomerates, promoting better conductivity. However, if the shear is too high, it can cause degradation of the binder or other sensitive materials. Conversely, lower speeds may lead to inadequate mixing, negatively impacting slurry viscosity and conductivity. Advanced mixing systems can be configured to provide tailored shear profiles, optimizing conductivity while maintaining material integrity.

Temperature

Temperature influences the viscosity and flow characteristics of the slurry. Optimizing temperature during mixing can enhance the solubility of binders and improve the dispersion of conductive additives. Higher temperatures can reduce viscosity, facilitating better flow and dispersion of materials. This improvement enhances contact between active particles and the electrical pathways within the slurry, ultimately boosting conductivity. Use of jacketed mixing vessels can provide effective temperature management throughout the process.

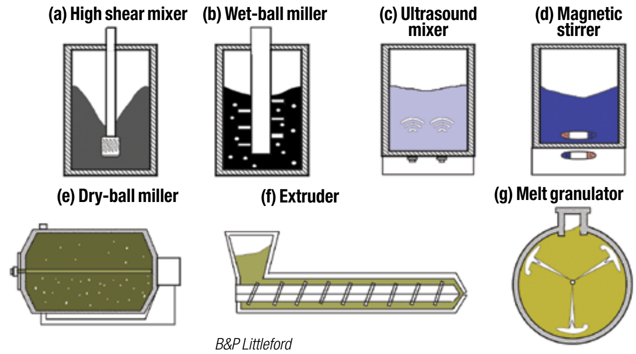

FIGURE 2. Several types of mixing equipment are available, categorized according to whether or not solvents or liquids are used to facilitate the mixing

Particle size distribution

The size and distribution of active materials and conductive agents in the slurry are key determinants of the battery electrode's ultimate performance. Smaller particles have increased surface area and ionic conductivity, but can also raise viscosity, complicating the mixing process.

Mixing technique

The choice between dry and wet mixing methods significantly affects material integration. Wet mixing, which involves the use of solvents or liquids to facilitate the mixing of active materials, conductive agents and electrolytes, often provides superior dispersion. Included in this category are ball milling, high-shear mixing and ultrasonic mixing. Dry mixing, on the other hand, involves blending solid powders without any liquid medium. It can lead to higher energy density by minimizing solvent content. Examples of this technique are tumbling mixers, ribbon blenders and planetary mixers (Figure 2).

The choice of mixing equipment, whether it is a planetary mixer, high-shear mixer, or dissolver, determines the intensity and type of forces applied to the slurry (Figure 3). Equipment that provides uniform shear forces is ideal for creating a well-dispersed slurry, leading to a more uniform electrode structure with lower resistivity and better performance.

The configuration of the mixing equipment is instrumental in determining mixing efficiency. Some systems utilize advanced geometries to enhance material interaction and ensure uniform dispersion of conductive additives. The ability to create a turbulent flow within the mixer helps break down agglomerates and promotes better conductivity throughout the slurry.

Also, the sequence in which the components are mixed affects the formation of conductive networks. For example, mixing the conductive agent with the solvent first, as opposed to simultaneously mixing all components, can result in a more stable and well-distributed conductive network. This method helps in preventing the collapse of the network structure during subsequent mixing stages, thereby improving the electrode's electronic properties.

Shear rate and viscosity

The intensity of shear forces during mixing influences particle distribution and orientation. Optimizing shear rates enhances mixing efficiency and slurry uniformity — critical factors for achieving consistent conductivity across the electrode.

The shear rate applied during mixing and the viscosity of the slurry are interconnected variables that influence the particle distribution and network formation within the slurry. These variables are closely related to shear stress, which plays a critical role in the mixing process.

The shear stress, τ, during mixing is defined by Equation (1):

$$\tau = 2.5 \cdot \gamma \cdot \eta \quad (1)$$

Where γ is the shear rate and η is the dynamic viscosity. A higher shear rate can improve the dispersion of particles within the slurry by breaking up agglomerates and promoting a uniform mixture. However, excessive shear can also lead to over-dispersion, where the conductive network becomes too fragmented, leading to higher resistivity. Viscosity affects how easily particles move and interact during mixing. A balance between shear rate and viscosity is essential to ensure optimal particle distribution without damaging the conductive network.

The shear number (*SN*), often a product of shear rate and time, quantifies the cumulative mechanical stress imparted to the slurry. It is expressed in Equation (2):

$$SN = (\gamma \cdot t) / (4 \cdot \pi) \quad (2)$$

where γ is the angular velocity and t is the mixing time. A high shear number can indicate extensive particle interaction and mixing, but excessive values can lead to over-dispersion and degradation of the electrode's structure.

The viscosity of the slurry must be carefully controlled to ensure effective mixing and pumping. High viscosity can lead to poor flow characteristics, which may result in uneven mixing and inconsistent conductivity. Utilizing specialized mixing equipment designed to handle varying viscosities can enhance material interaction and ensure a uniform slurry. Monitoring viscosity in real-time allows for adjustments during processing to maintain optimal flow.

Composition and materials

The electrical conductivity of the battery slurry is significantly influenced by the characteristics of its components. Active materials, such as lithium cobalt oxide or lithium iron phosphate, must be optimally blended with conductive additives like carbon

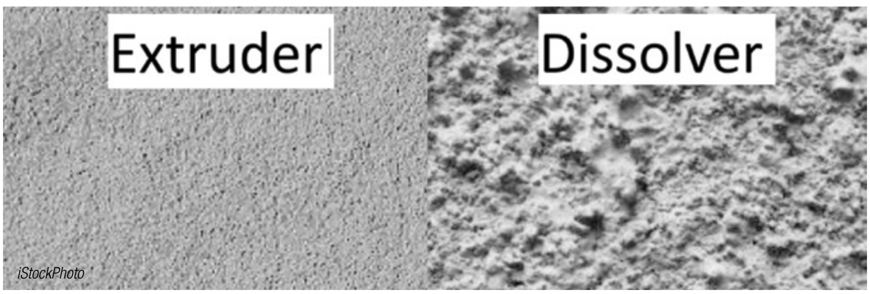

FIGURE 3. The micrographs illustrate the degree of dispersion in the electrode slurry. At left, the dispersion achieved with an extruder is higher than that observed with a wet-mixing method

black or graphene. The choice and quality of these materials determine the intrinsic conductivity of the slurry. For example, the surface area of carbon black directly correlates with its conductivity; thus, using high-performance conductive additives can enhance the overall conductivity of the battery slurry.

The slurry composition, including the proportions of active material, conductive agents, binders and solvents, plays a crucial role in determining the final properties of the electrode and in controlling the conductivity. A balanced composition of these components enhances electron transport and ensures sufficient conductivity without compromising the mechanical properties of the slurry. Excessive concentrations can negatively impact the mechanical properties and conductivity. This is due to increased particle-particle interactions, leading to agglomeration. On the other hand, too little of the active material and conductive agents may not provide the desired conductivity. Process optimization based on empirical data can help identify the best ratios for specific slurry formulations.

The distribution of the active material (for example, graphite for the anodes) and the conductive agents (such as carbon black) within the slurry is critical. Uniform dispersion ensures effective electrical conductivity throughout the electrode. Poor dispersion can lead to the formation of agglomerates, which increase electrical resistivity and reduce the battery's overall performance. Moreover, the order in which these components are mixed significantly affects the dispersion of the conductive materials within the slurry.

The solvent-to-solids ratio in wet mixing also affects the slurry's viscosity and particle mobility. Achieving an optimal liquid-to-solid ratio in the slurry is vital for ensuring sufficient dispersion without excessively diluting active materials. ■

Edited by Scott Jenkins

Authors

Janina Lazo-Cruz is a versatile article writer, covering a wide range of topics while actively pursuing a career as a process engineer at a leading Japanese multinational company in electronic manufacturing. A chemical engineer and materials scientist by training, she graduated from the University of the Philippines and has a keen interest in data analytics, lean manufacturing, R&D and product development, and polymer chemistry. She can be reached at the following Email address: janinalazo@gmail.com.

Daniel Shafer is responsible for field application engineering on industrial scale chemical process equipment on behalf of B&P Littleford (1000 Hess Ave., Saginaw, MI 48601; Email: dshafer@bplittleford.com; Website: www.bplittleford.com). Shafer serves as global sales manager in the Battery Materials Division and is western U.S. sales manager for the entire line of B&P Littleford equipment. Shafer serves as co-director of Nikua Training Center, a multinational non-profit company promoting energy security in the South Pacific. Shafer holds a degree in chemical engineering from Oregon State University.

Automation Optimizes Rinsewater Recycling

Onsite recycling proved cost-effective for an aerospace supplier. With real-time feedback and control, a new automation upgrade takes process efficiency to the next level

Rick Hines
GF Piping Systems

Throughout the chemical process industries (CPI), efficient and affordable approaches to wastewater treatment are of paramount importance. In particular, onsite water recycling can support the bottom line while furthering sustainability goals. Ion-exchange systems offer a proven, economically viable technology for recycling industrial wastewater with high levels of recovery. While the initial equipment costs of this approach can be significant, they are more than outweighed by the long-term economic and environmental benefits. At the same time, system designers are always seeking even greater cost-effectiveness. As Ethernet-enabled components become ever more sophisticated, automation can provide this crucial edge.

Building on success

In 2017, a manufacturer in the aerospace sector worked with Water Innovations, Inc. (San Diego, Calif.; www.waterinnovations.net) to develop an onsite system for closed-loop recycling of its plating rinsewater. For more on this project, read Cutting Cost and Waste with Rinsewater Recycling, *Chem. Eng.*, May 2023, pp. 45–46. The company's operations require a high volume of deionized water (DI) to rinse metal parts as they are cleaned, etched and electrolytically plated with specialized metal finishes. To address both their supply and treatment needs, Water Innovations designed and installed three different resin-based ion exchange systems as well as a bentonite reactant treatment system for the oiliest waste. GF Piping Systems (GF; Irvine, Calif.; www.gfps.com), a longtime project partner for Water Innovations, provided crucial components and expertise.

Overall, the manufacturer's total wastewater management costs dropped by 80%, with a 60% reduction in treated effluent going to the sewer. The onsite recycling system furthermore yielded an abundant supply of DI water, with cost per gallon reduced by 70%. After only two years of operation, the company achieved return on its Water Innovations investment. Then, in 2023, Water Innovations saw the potential to build even further on this success.

FIGURE 1. An automation upgrade using the EtherNet-equipped EA-25 electric actuator interface allowed an aerospace manufacturer to achieve greater efficiency for its onsite recycling process

For more than twenty years, Water Innovations systems have utilized "feed-forward" grain counting to trigger countercurrent regeneration just before the resin beds became exhausted — instead of just after. Countercurrent (or reverse-flow) regeneration provides efficiency advantages in and of itself, on the order of 50–60% versus co-current methods. This is because the regenerant solution comes into contact with the less-exhausted resin layers first, so that a lower quantity of solution is ultimately needed. In recent years, Water Innovations has assessed contaminant loading in rinsewater by measuring its conductivity, using sensors provided by GF. This additional sophistication (as opposed to simple assessment via pH) already cuts the amount of wastewater sent to treatment by as much as 20%. But now, the advent of an Ethernet-enabled actuator (Figure 1) from GF offered a chance to streamline the regeneration process with even greater efficacy.

As originally designed, the aerospace manufacturer's Water Innovations system relied on GF Type 523 Metering Ball Valves, which have a 180-deg rotation V-notch design for greater resolution and linearity, to adjust the dosing of the regenerant solution. Operators would calculate the required chemical concentration and then manually adjust the valves to deliver the desired dose. Water Innovations designed an upgrade for the system, known as Optimized Regeneration Control (WI-ORC), in which the dosing would be monitored and adjusted automatically in real time. With automatic adjustments based on constant feedback, the time and materials required for regeneration could be minimized.

Real-rime precision cuts costs

As part of this upgrade, GF provided its newest electric actuator, the Type EA25-250, equipped with Ethernet/IP communications via M12

field connections. Other key components included GF's 3-2823 conductivity sensors and 9950 dual-channel controller, providing an expanded conductivity range up to 800 mS/cm for measuring the individual chemical concentrations of the regenerant chemicals hydrochloric acid (HCl) and sodium hydroxide (NaOH).

In the upgraded system, automation serves to optimize the results. GF conductivity sensors monitor the concentrations of HCl and NaOH. The dual-channel transmitter sends the conductivity values to the WI-ORC system, where an algorithm calculates proportional-integral-derivative (PID) setpoints that can be transmitted to the electric actuator through its Ethernet/IP capability. The actuator and metering valve are thus continuously positioned to maintain an optimal chemical dose throughout the resin regeneration process. The Water Innovations control system is capable of continuously monitoring the actuator's performance, including such parameters as valve position (%), cycle time, motor torque, total cycles and internal temperature, as well as providing detailed error flags. With the deployment of the web server, managers can externally configure the electric actuator's open/closed end points, or select the desired color scheme for open and closed status (either red for closed and green for open or vice versa). The actuator can also receive firmware updates.

With the real-time precision of automation, the time required for resin regeneration has been cut by 10%. The amount of regeneration chemicals needed and the amount of regeneration wastewater sent to the municipal waste-treatment system have each been reduced by 10% as well. Factoring in the reduced outlay for chemicals and discharge fees, the upgrade is expected to achieve return on investment in just 15 months. Beyond the cost savings, the automated system's performance is also more consistent, continuously providing high-quality water without dependence on the operator.

"We're always looking for ways to fine-tune systems we've already built, even highly efficient ones. In this case, the Ethernet capability of GF's new actuator let us take that next step," said Steven Ward, managing partner at Water Innovations. With continual measurement and adjustment in real-time, the potential of automation to optimize process costs and outcomes has only just begun to be realized. ■

Edited by Mary Page Bailey

Photo courtesy of GF Piping Systems

Author

Rick Hines is the director of automation for GF Piping Systems' U.S. Center of Excellence (9271 Jeronimo Rd., Irvine, CA 92618; Email: rick.hines@georgfischer.com). With over four decades of experience in the field of fluid handling, Hines is active in the effort to provide safe, clean and reliable water supplies to communities throughout the world.

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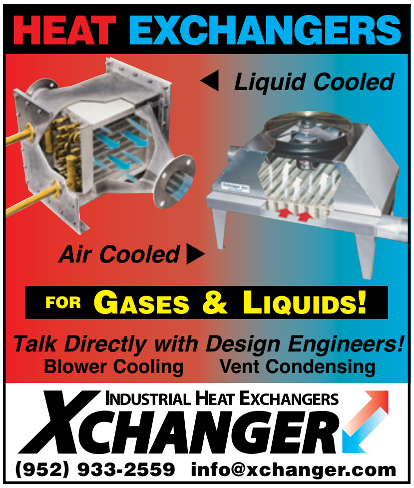

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North America

Matthew Grant | **VP & Group Publisher**
mgrant@accessintel.com

Terry Davis | **District Sales Manager**
Tel: 404-481-0254
E-mail: tdavis@accessintel.com
Chemical Engineering 2276 Eastway Rd., Decatur, GA 30033
Connecticut, District Of Columbia, Delaware, Georgia, Maine, Maryland, Massachusetts, New Hampshire, New Jersey, New York, Pennsylvania, Rhode Island, Vermont, Canada, Latin America

International

Petra Trautes
Tel: +49-172-6606303
Email: ptrautes@accessintel.com
Access Intelligence International Postfach 20 01 39
D-63307 Roedermark
Europe

Dipali Dhar
Tel: 917-592-7729
E-mail: ddhar@accessintel.com
Chemical Engineering 40 Wall Street, 16th Floor, New York, NY 10005
India

Katshuhiro Ishii
Tel: 81-3-5691-3335
Fax: 81-3-5691-3336
E-mail: amskatsu@dream.com
Chemical Engineering Ace Media Service Inc., 12-6, 4-chome Nishiiko, Adachi-ku, Tokyo 121, Japan
Japan

Rudy Teng
Tel: +86 13818181202, (China)
+886 921322428 (Taiwan)
Fax: +86 21 54183567
E-mail: rudy.teng@gmail.com
Chemical Engineering 8F-1
#181 Wulin Road Hsinchu 30055 Taiwan
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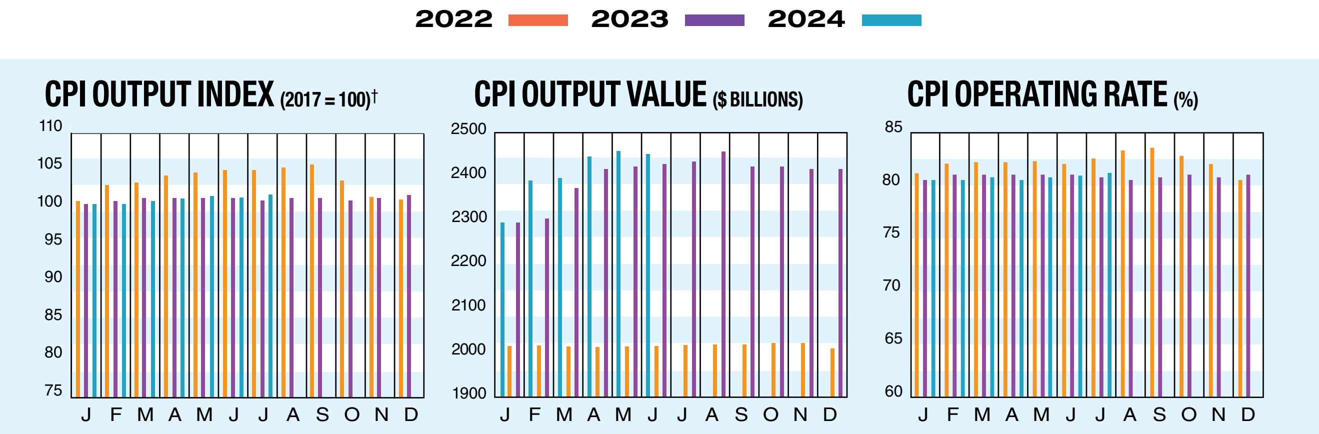
CPI output index (2017 = 100)	Jul. '24	=	100.9	Jun. '24	=	100.2	May '24	=	100.3	Jul. '23	=	99.3
CPI value of output, $ billions	Jun. '24	=	2,414.5	May '24	=	2,421.7	Apr. '24	=	2,461.8	Jun. '23	=	2,321.3
CPI operating rate, %	Jul. '24	=	77.6	Jun. '24	=	77.2	May '24	=	77.4	Jul. '23	=	77.8
Producer prices, industrial chemicals (1982 = 100)	Jul. '24	=	309.5	Jun. '24	=	303.2	May '24	=	302.0	Jul. '23	=	309.8
Industrial Production in Manufacturing (2017 =100)*	Jul. '24	=	99.5	Jun. '24	=	99.8	May '24	=	99.8	Jul. '23	=	99.4
Hourly earnings index, chemical & allied products (1992 = 100)	Jun. '24	=	228.8	May '24	=	228.4	Apr. '24	=	226.4	Jun. '23	=	222.6
Productivity index, chemicals & allied products (1992 = 100)	Jul. '24	=	93.1	Jun. '24	=	93.1	May '24	=	93.1	Jul. '23	=	94.3



*Due to discontinuance, the Index of Industrial Activity has been replaced by the Industrial Production in Manufacturing index from the U.S. Federal Reserve Board.
†For the current month's CPI output index values, the base year was changed from 2012 to 2017
Current business indicators provided by S&P Global Market Intelligence, New York, N.Y.